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LOX-LUBRICANT IMPACT SENSITIVITY RESULTS FOR ASD COOPERATIVE TEST PROGRAM NO. 3

SPECIAL REPORT
Contract AF 33(616)-7223
Project No. 3044
Task Nos. 304401 and 304402

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to

Aeronautical Systems Division
Wright-Patterson Air Force Base, Ohio

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FOREWORD

This report was prepared at Southwest Research Institute under USAF Contract AF 33(616)-7223. The contract was initiated under Project No. 3044, "Aerospace Lubricants," Task No. 304401, "Turbojet Engine Lubricants," and Task No. 304402, "Missile and Space Vehicle Propulsion Lubricants." The work was administered by the Nonmetallic Materials Laboratory, Directorate of Materials and Processes, Aeronautical Systems Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The project engineer was Mr. G. A. Beane.

This report covers work performed by ten cooperating laboratories in the period of November, 1960 through December, 1962.

ABSTRACT

The results from LOX-Lubricant Impact Sensitivity Cooperative Program No. 3 are presented. Impact sensitivity threshold values of three test samples were evaluated by ten laboratories. Seven laboratories reported results using the ABMA type impact tester and three laboratories reported results using the RMD impact tester. Even though rather large variations in specific threshold values were reported by laboratories using the ABMA type impact tester, there was general agreement between laboratories with respect to the relative sensitivity of the three test samples evaluated. General agreement of relative sample sensitivity was also obtained by laboratories using the RMD impact tester; however, the order of sample sensitivity was different from that obtained with the ABMA type impact tester.

Although the LOX-Lubricant Impact Sensitivity Cooperative Program No. 3 showed definite improvements in test reproducibility from the previous cooperative test programs, further improvement in test reproducibility still appears desirable.

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I. INTRODUCTION

This report presents the results obtained by ten cooperating laboratories from the LOX-Lubricant Impact Sensitivity Cooperative Test Program No. 3. The program was initiated by ASD in an effort to establish the reproducibility of impact sensitivity threshold value determinations using the ABMA type impact tester and to determine the degree of correlation of results obtained using the RMD impact tester with those obtained using the ABMA type impact tester.

Two impact test cooperative programs[1, 3]* were conducted prior to the present program. In the first cooperative program, completed in 1958, no attempt was made to standardize the equipment used by the nine participating laboratories and "Most of the testers differed with respect to sample cups (foil, welded, stamped), striker pin area (0.033 to 1.77 sq in.) or plummet weights (1.1 to 70 lbs)"[1]. The results obtained from Cooperative Program No. 1 indicated very poor agreement of data from the various testers. The major conclusion from the program was that there was a need for standardization of equipment and test method. The second cooperative program, conducted in 1960, incorporated a number of significant improvements in the test procedure and equipment[2]. The data obtained from this program indicated an improvement in the correlation of results from the different laboratories[3]. However, it was agreed at a meeting of the cooperating laboratories that while an improvement in correlation of results was obtained, the degree of correlation was not as good as desired. Thus a third cooperative program, with more stringent test procedure and equipment requirements, was initiated.

II. PARTICIPATING LABORATORIES

The laboratories that participated in the Cooperative Test Program No. 3 and the testers used by the participants were as follows:

Laboratory**	Tester Type
Air Force Flight Test Center	ABMA
General Dynamics/Astronautics	"
Marshall Space Flight Center	"
Monsanto Research Corp.	"
North American Aviation, Rocketdyne Division	"
Pratt & Whitney Aircraft, FRDC	"
Southwest Research Institute	"
Douglas Aircraft	RMD
Minnesota Mining & Manufacturing Co.	"
Thiokol Chemical Corp., Reaction Motors Division	"

* Superscript numbers in parenthesis refer to List of References.

** With the exception of Southwest Research Institute (SwRI), each participation laboratory is hereinafter designated by a leter code.

It was originally planned that SwRI would also conduct cooperative tests using an RMD tester supplied by the manufacturer. However, repeated attempts by SwRI and the manufacturer to calibrate the tester installed at SwRI within the accuracy limits reported for the tester were unsuccessful. Consequently, SwRI did not conduct further tests with the RMD tester. A detailed report of the calibration attempts on the RMD tester at SwRI is presented in Appendix IV.

III. TEST SAMPLES

The following three fluid samples were selected for use in Cooperative Test Program No. 3:

Sample Code	Identification
III A	Dow Corning QF-6-7012
III B	D.C. 510, 500 cs
III C	Aroclor 1254

Sufficient quantities, from single batches, of each of the fluid samples were obtained and distributed to the cooperating laboratories as directed by ASD.

IV. TEST EQUIPMENT

A. ABMA Type Impact Tester

The ABMA type impact tester consists of a 20-lb plummet assembly which is guided in its vertical travel by three guide rails. Friction between the plummet assembly and the guide rails is minimized by the use of six small rollers, three each attached to both the upper and lower spider plates of the plummet assembly. The plummet assembly may be held at any desired vertical height, between 0 and 50 in., by means of an electromagnet, which is clamped to a fourth rail and whose position on the fourth rail is adjustable. The plummet may be released by de-energizing the magnet, whereupon it is allowed to drop upon a striker pin previously placed in position in a specimen cup containing the test sample and the oxidizer. A detailed description of the ABMA type impact tester is presented in the USAF Specification Bulletin 527 which is included in this report as Appendix I.

The anvil region assembly described in USAF Specification Bulletin 527 was supplied to six of the seven laboratories by SwRI at cost. The seventh laboratory reported that the assembly would be made in their machine shop using the drawings supplied by SwRI. Therefore, it is

assumed that all laboratories reporting results from the ABMA type impact tester used the standard tester described.

B. RMD Impact Tester

The operating principle of the RMD impact tester is identical to that of the ABMA type tester The RMD tester consists of a 2000-gm (approximately 4 41 lb) plummet assembly which is guided in its vertical travel by three ground rods Friction between the plummet and the guide rods is minimized by two low friction rings on the plummet bearing surface. The plummet assembly may be held at any desired vertical height, between 0 and 48 in , by means of a spring-loaded latch attached to a plummet release platform, which is clamped to one of the three guide rods and whose position on the rod is adjustable for height. The plummet may be released by energizing a solenoid coil which withdraws the spring-loaded latch thus permitting the plummet to drop upon the vented plug, cap, and die cup assembly containing the test sample and oxidizer.

A detailed description of the RMD tester is presented in Appendix III, a reproduction of the "Impact Tester Operating Instructions" furnished by the manufacturer with the tester.

C Comparison of Impact Testers

The principal differences in the two impact testers may be readily compared in the following tabulation:

Item	Impact Tester	
	ABMA	RMD
Plummet weight, lb	20	4.41
Striker pin diameter, in	0.50	0.201
Striker pin areas, sq in	0.196	0.032
Specimen cup cap	not required	scalloped Al (0 012 in thick)
Specimen cup diameter, in	1.0	0.355
Sample size required, ml	0 50 to 0.60	0.008 to 0.009

D. Striker Pins and Specimen Cups

1. ABMA Type Impact Tester

The striker pins and specimen cups used in this program with all the ABMA type impact testers were standard items (see Appendix I) from one batch of pins and cups received from the suppliers by SwRI. The striker pins were individually inspected for dimensions and surface finish at SwRI. The surface finish, measured with a Brush Surfindicator, varied between 60 to 70 microinches RMS for all the striker pins used in this program. Random inspection (1 in 10 cups) was made at SwRI on the specimen cups supplied to the cooperating laboratories.

It is of interest to note that Laboratory C commented that "the specimen cups provided for this program were found to have an abnormal amount of a black film contaminant on the inner surfaces which necessitated additional cleaning cycles for complete removal of contaminant," compared to other batches of specimen cups from the same manufacturer. Upon checking with the manufacturer, it was found that the black film contaminant was a die lubricant used in the manufacturing process. According to the manufacturer, some of the earlier batches of specimen cups had been rinsed at the factory with a solvent to remove this film; however, the particular batch supplied for the cooperative program did not receive the solvent rinse at the factory.

The lubricant film contaminant (black film) on the specimen cups was also noted by SwRI; however, it did not appear abnormal since SwRI has never received specimen cups which were rinsed at the factory before delivery. It has been general practice at SwRI to remove the lubricant film by wiping the specimen cups with a shop towel while visually inspecting each specimen cup prior to starting the required cleaning procedure.

2. RMD Impact Tester

It is assumed that standard RMD die cup assemblies were used by the laboratories reporting results from the RMD tester since no deviations were noted

V TEST PROCEDURE

A. ABMA Type Tester

A tentative test procedure to be used with the ABMA type impact tester was discussed at the November 17, 1960 meeting of the cooperating laboratories. This procedure, with minor changes, was later published by ASD as USAF Specification Bulletin 527 (Appendix I). A copy of Bulletin 527 was sent to each cooperating laboratory, and it was requested that all laboratories use the procedure described therein. Later a modified precooling procedure (see Appendix II) to replace paragraph 6.2.3.2 of Bulletin 527 was distributed to each laboratory in an effort to provide a definite sequenced precooling procedure.

B. RMD Tester

The original test procedure recommended for use with the RMD tester was the procedure described in Appendix III. However, a revised procedure described in RMD Specification 7491, "Procedure for Operating the Reaction Motors Impact Tester, RMD P/N 311636," dated September 1, 1961, was later recommended. The revised procedure is essentially

the same as that described in Appendix III except that the position of the slot in the vented plug with respect to the centerline of the scalloped cap is defined (see Appendix IV).

VI TEST DATA REQUESTED

It was requested that each laboratory perform twenty test drops on each test sample at drop heights of 42, 33, 24, and 15 inches and that a definitive threshold value (paragraph 8.7.2 of Specification Bulletin 527) be obtained for each of the test samples.

In addition to the above test data, it was requested that an estimate of the intial rebound height of the plummet during blank test be reported.

VII TEST RESULTS AND DISCUSSION

A. General

Summaries of the impact test data submitted by the laboratories using the ABMA type impact tester and the RMD impact tester are presented in Tables 1 and 2, respectively. As indicated in Table 1, Laboratory D did not obtain a definitive threshold value for sample III A and Laboratory E did not obtain definitive threshold values for samples III A and III C. If a strict interpretation of the definition of definitive threshold value is taken, there are additional reported threshold values which may be suspect. The definitive threshold value of a sample material is defined in Specification Bulletin 527 as "the potential energy level for the higher of the two highest adjacent drop heights at which no reaction occurred in 20 drops, and below which level no reaction occurred." This particular point is not believed to be serious if the threshold value is determined to be 12 in., since the tester is not usually operated at drop heights of less than 12 inches. However, in the cases of the threshold values reported by Laboratory C for sample III A, and Laboratories A, C, and E for sample III B, it should be pointed out that no reactions in 20 test drops at one drop height may produce erroneous indications of a threshold value as evidenced by the following data taken from Table 1:

Drop	Reaction Frequency			
	Sample III A		Sample III B	Sample III C
Hgt., in.	SwRI	D	SwRI	B
27	-	-	1/14	4/20
24	1/20	1/20	0/20	0/20
21	0/20	0/20	1/6	2/20
18	1/20	1/20	0/20	0/20
15	0/20	1/20	0/20	0/20
12	0/20			

TABLE 1. SUMMARY OF IMPACT SENSITIVITY TEST RESULTS FROM LABORATORIES USING THE ABMA IMPACT TESTER

Drop Hgt., in.	Reaction Frequency, No. of Reactions/No. of Test Drops																									
	Sample III A							Sample III B							Sample III C											
	SwRI	A	B	C	D	E	F	SwRI	A	B	C	D	E	F	SwRI	A	B	C	D	E	F					
48													1/10							2/20						
45													5/11													
42	4/20	1/3	9/20	5/20	2/20	5/10	5/20	5/20	1/2	8/20	1/20	1/20	0/20	3/20	2/20	1/5	5/20	1/20	3/20	6/20	2/20					
39											0/20															
36																										
33	4/20	1/1	11/20	2/20	2/20	4/6	2/20	3/20	1/8	3/20	0/20	2/22		1/20	1/20	1/8	6/20	3/20	4/20	3/20	1/20					
30				1/20			2/4	1/5	1/5		0/20			0/20							0/20					
27				2/20			0/20	1/14	1/1					0/20			4/20				0/20					
24	1/20	1/3	3/20	0/20	1/20	3/10	0/20	0/20	0/20	1/20	0/20	2/20		0/20	1/20	1/2	0/20	1/20	3/20	4/20	0/20					
21	0/20				0/20			1/6		1/20		1/20			1/6		2/20									
18	1/20				1/20			0/20		0/20		0/20			1/2		0/20									
15	0/20	1/9	2/20	0/20	1/20	3/16	0/20	0/20		0/20	0/20	0/20		0/20	0/20	1/2	0/20	1/20	3/19	4/20	0/20					
12	0/20	0/20	0/20												0/20	1/6		0/20	1/20							
9																0/20			1/20							
6																		0/20	2/20							
3																			0/20							

TABLE 2. SUMMARY OF IMPACT SENSITIVITY TEST RESULTS FROM LABORATORIES USING THE RMD IMPACT TESTER

	Reaction Frequency, No. of Reactions/No. of Test Drops					
	Sample III A		Sample III B		Sample III C	
Drop Hgt., in.	G	H	G	H	G	H
42		5/6				9/10
41						
40					7/20	
39						
38						
37						
36						
35						
34					6/20	
33		4/10		3/3		6/10
32						
31						
30						
29						
28	7/20		13/20		5/20	
27						
26						
25						
24		5/10		8/10	3/20	3/11
23						
22					1/20	
21						
20	4/20		7/20		0/20	
19						
18						
17						
16	3/20					
15	2/20	4/10		2/10		2/10
14	0/20	1/7	6/20	1/10		0/10
13		1/4				
12		1/1	2/20			
11		1/6	1/20			
10		1/1	0/20			
9		0/9				

No detailed test results reported by Laboratory I.

Laboratory H, using an RMD tester, reported a threshold value of 14 in. on sample III B. However, the data sheets submitted listed one reaction, obtained on rebound, during the ten test drops at 14 in. thereby indicating that the threshold value is below 14 inches.

The threshold values, taken from Tables 1 and 2, for the three test samples are tabulated in Table 3 for the ABMA type tester and Table 4 for the RMD tester. The threshold values are also presented graphically in Figures 1 and 2 for easy reference.

It will be noted in Figure 1 that although there is a large spread in the individual threshold values reported for the three test samples, there is general agreement between five of the seven reporting laboratories with respect to the relative sensitivity of the three test samples. Further, all seven laboratories agree with respect to the relative sensitivity of samples III A and III B.

Exceptionally good agreement of threshold values is indicated in Figure 2 by two of the laboratories using the RMD tester. It is possible that better overall agreement of relative sensitivity of the three test samples may have been obtained if more complete data were available from Laboratory H on sample III B.

Comparing the results obtained using the two different testers, it is of particular interest to note that when the threshold values obtained with the ABMA type tester (Fig. 1) are compared with those obtained with the RMD tester (Fig. 2), a different order of relative sensitivity of the three test samples is indicated. The ABMA type tester, in general, rated sample III C as the most sensitive and sample III B as the least sensitive, while the RMD tester rated sample III C as the least sensitive and sample III B as the most sensitive of the three samples tested.

There was considerable discussion at the November 1960 meeting of the cooperating laboratories on the validity and identification of char marks as evidence of reactions in the event that no other indication of a reaction was noticeable during the test drop. With this in mind, the data submitted for Cooperative Program No. 3 was reviewed. Laboratory C reported only one char mark and Laboratories D and F did not report any char marks. SwRI and Laboratories A, B, and E reported a char mark in conjunction with practically every audible reaction recorded. However, very few char marks were reported without additional evidence of reactions occurring, and none of the threshold values recorded (Table 3) were based upon char reactions alone.

B. Supplementary Data

Any investigation of the reproducibility of a test method should be judged in the light of the repeatibility of the test method. Table 5 presents

TABLE 3. SUMMARY OF THRESHOLD VALUES FROM LABORATORIES USING THE ABMA IMPACT TESTER

	Threshold Value								
	Sample III A			Sample III B			Sample III C		
Laboratory	in.	ft-lb	ft-lb/in.^{2}	in.	ft-lb	ft-lb/in.^{2}	in.	ft-lb	ft-lb/in.^{2}
SwRI	15	25	128	18	30	153	15	25	128
A	12	20	102	24	40	204	9	15	77
B	12	20	102	18	30	153	18	30	153
C	24	40	204	39	65	332	12	20	102
D	<15	<25	<128	18	30	153	3	5	26
E	<15	<25	<128	42	70	357	<15	<25	<128
F	27	45	230	30	50	255	30	50	255

TABLE 4. SUMMARY OF THRESHOLD VALUES FROM LABORATORIES USING THE RMD IMPACT TESTER

	Threshold Value					
	Sample III A		Sample III B		Sample III C	
Laboratory	in.	ft-lb/in.^{2}	in.	ft-lb/in.^{2}	in.	ft-lb/in.^{2}
G	14	162	10	116	20	231
H	9	104	<14	<162	14	162
I	14	161*	12	138*	19	217*

*These data were the only information reported. The corresponding inch values were obtained by dividing reported data by $4.41/(0.032 \times 12) = 11.55$ and rounding off to the nearest inch.

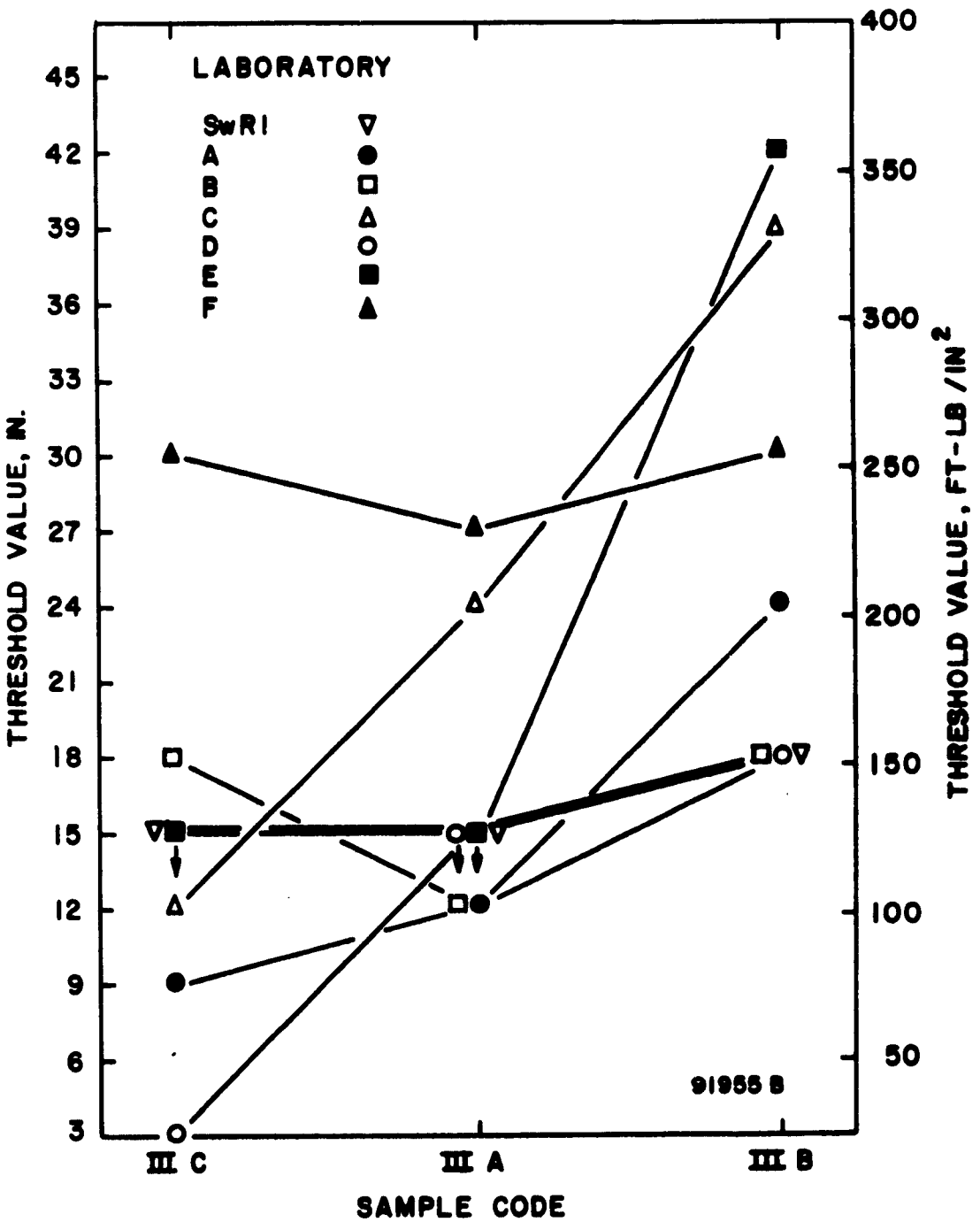

FIGURE 1. THRESHOLD VALUES REPORTED BY LABORATORIES USING THE ABMA IMPACT TESTER

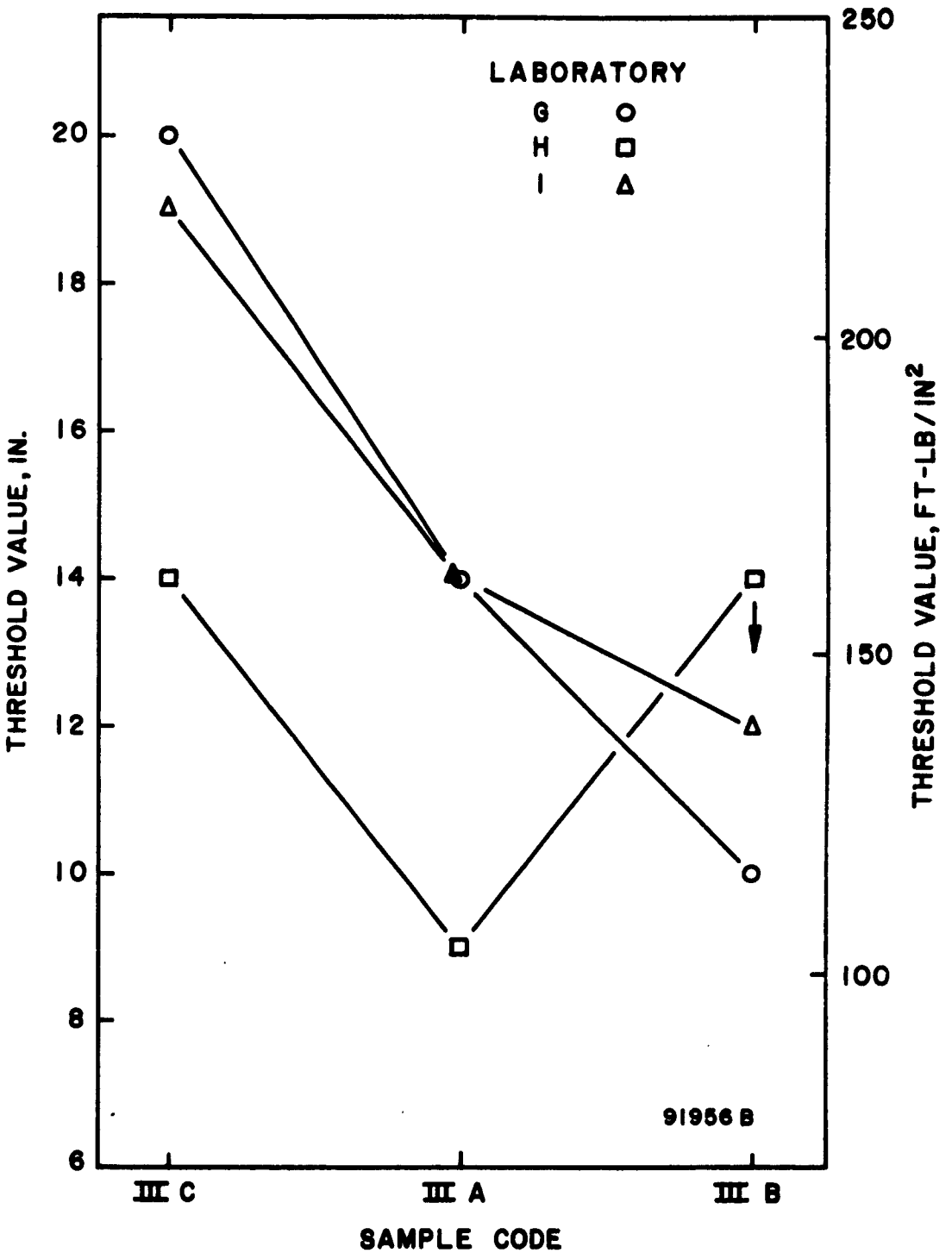

FIGURE 2. THRESHOLD VALUES REPORTED BY LABORATORIES USING THE RMD IMPACT TESTER

TABLE 5. SUMMARY OF THRESHOLD VALUES OBTAINED BY SwRI ON COOPERATIVE TEST PROGRAM NO. 3 TEST SUPPLIES

Sample	Date of Test	Threshold Value	
		Height, in.	Potential Energy, ft-lb
III A	May 1961	21	35
	May 1961	18	30
	June 1961	15	25
	June 1961	15	25
	June 1961	18	30
	June 1961	18	30
	Oct. 1961	15	25
	Oct. 1961	12	20
	Oct. 1961	12	20
	Oct. 1961	18	30
	Nov. 1961*	15	25
	Nov. 1961	18	30
	Nov. 1961	21	35
	Dec. 1961	21	35
	Dec. 1961	18	30
	July 1962	18	30
III B	June 1962	18	30
	June 1961	30	50
	June 1961	18	30
	June 1961	27	45
	July 1961	24	40
	July 1961	30	50
	Aug. 1961	27	45
	Nov. 1961*	18	30
III C	Mar. 1961	18	30
	May 1961	12	20
	May 1961	15	25
	June 1961	18	30
	Nov. 1961*	15	25

*These data were obtained and reported for Cooperative Test Program No. 3.

the threshold values obtained at SwRI on the samples used in Cooperative Test Program No. 3, by means of the ABMA impact tester using the test method specified in Specification Bulletin 527. A summary of these threshold values is as follows:

Sample	Threshold Value, in.			
	Minimum	Maximum	Average	Standard Deviation
III A	12	21	17.0	2.84
III B	18	30	24.0	5.32
III C	12	18	15.6	2.51

Figure 3 presents a comparison of the threshold values reported for Cooperative Test Program No. 3 with those obtained in the repeatibility study. The range of threshold values determined in the repeatibility study is represented by the shaded area. It will be noted that the majority of the threshold value determinations reported for the present cooperative program are within the repeatability range of one laboratory.

C. Test Variations Reported

1. Test Procedure

The only variation in the test procedure used was reported by Laboratory B; "Liquid nitrogen instead of LOX was used for precooling strikers, cups, and anvil similar to the method used for Cooperative Program No. 2."

2. Sample Volume

A variation in the amount of test sample used to attain the required 0.050 in. sample thickness was noted in the data submitted. Following is a tabulation showing the different amounts of test sample used:

Laboratory	Sample Used, ml		
	III A	III B	III C
SwRI	0.57	0.60	0.56
A	0.55	0.55	0.46
B	0.55*	0.55*	0.53*
C	0.50	0.50	0.50
D	Not Reported		
E	0.50	0.50	0.50
F	0.60	0.60	0.60

* Approximate values calculated from weights reported.

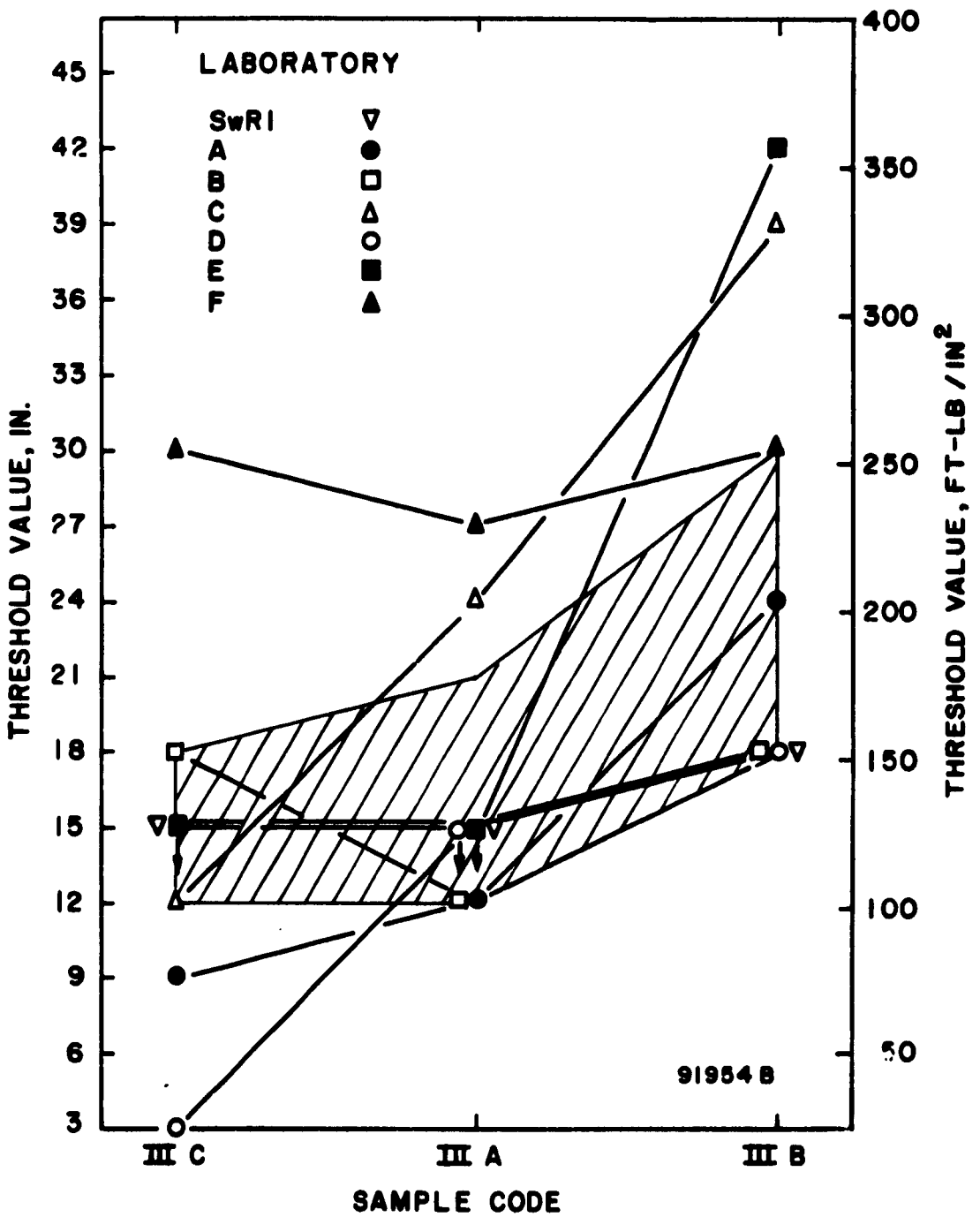

FIGURE 3. COMPARISON OF REPORTED THRESHOLD VALUES WITH THOSE OBTAINED BY ONE LABORATORY USING THE ABMA IMPACT TESTER

Using the minimum and maximum reported quantities of each test sample, measurements taken at SwRI indicate that the sample thicknesses used by the different laboratories may have varied between approximately 0.040 and 0.054 in. for all three test samples. However, based upon the threshold values reported by the cooperating laboratories and upon data obtained earlier at SwRI on the effect of sample thickness on sensitivity[4], it is not believed that this apparent discrepancy is significant in the present program.

3. Plummet Drop Time

The reported plummet drop time variations are tabulated in Table 6 as percent variation from calculated free fall. Four laboratories report consistent average drop times within the allowable 3 percent variation.

4. Rebound Height

The following initial rebound heights of the plummet were reported for blank tests conducted at 48 in. drop height on the ABMA type testers (in the order of decreasing rebound height):

Laboratory	Rebound Height, in.
A	24
SwRI	9
E	6 to 12
F	6 to 8.5
B	4.7*
D	3**
C	.75 to 1.25

It was believed that the severity of the individual impact testers could be indicated by comparing the rebound heights and the threshold values obtained with the various testers. However, it can be seen in the following tabulation that although there are indications that some testers are generally more severe (for example, A, D, and SwRI) than others, the test severity does not appear to be consistently related to the rebound height.

	Relative Severity		
Order of Severity	III A	III B	III C
Most severe	A, B, D, E	SwRI, B, D	D
↓	SwRI	A	A
	C	F	C
	F	C	E
		E	SwRI
			B
Least severe			F

* Reported calculation from data at 24 and 36 in. drop heights.
** Data reported for 42 in. drop height.

TABLE 6. SUMMARY OF AVERAGE VARIATION OF DROP TIME FROM CALCULATED FREE FALL

Laboratory	Sample	Calculated Mean Variation from Free Fall, percent 42 in	33 in.	24 in.	15 in.
SwRI	III A	1.8	1.7	1.9	2.3
	III B	1.3	1.4	1.7	2.3
	III C	1.8	1.5	1.5	2.5
A	III A	2.7(3)	3.6(1)	4.6(3)	1.5(9)
	III B	3.0(2)	2.6(8)	1.8	-
	III C	2.9(5)	3.0(8)	2.8(2)	1.8(2)
B*					
C	III A	0.9	1.2	2.4	2.3
	III B	1.5	1.2	2.0	2.4
	III C	1.0	1.1	2.5	2.4
D	III A	0.6	2.0	1.2	0.4
	III B	0.6	1.9	0.8	1.1
	III C	0.2	1.7	1.9	1.3
E	III A	2.9(10)	4.1(6)	4.3(10)	4.2(16)
	III B	2.0	-	-	-
	III C	3.4	3.8	3.3	4.0
F**	III A	0.4	0.5	0.0	-0.4
	III B	-0.6	0.0	-0.3	0.7
	III C	-0.4	-0.5	-0.3	0.7

Values given are based upon 20 test drops unless indicated by number in parenthesis which denotes number of drops used to obtain mean.

* No timing data submitted.

** Corrected drop time (average recorded drop time minus 20 milliseconds) reported. "The cause for this deviation of measured from theoretical free fall time has been found in the continuing support of the plummet by the decaying magnetic field after the plummet has moved far enough to break the timer contacts. This deviation has been found to be systematic, and under the specified conditions of operation, constant at 20 ± 2 milliseconds."

VIII. CONCLUSIONS

Despite the rather large variations in the threshold values reported by laboratories using the ABMA type impact tester, there was general agreement among laboratories with respect to the relative sensitivity of the three test samples evaluated in the program.

General agreement of relative sample sensitivity, in a different order from that obtained with the ABMA type tester, was indicated by the laboratories reporting results from the RMD impact tester.

No direct correlation of results from the two different testers was evident except to the extent that both testers showed all three test samples to be impact sensitive in LOX.

Cooperative Test Program No. 3 showed definite improvement in test reproducibility when compared with previous cooperative test programs. However, further improvement in test reproducibility is desirable. It is recommended that additional emphasis be placed on the control of variables such as sample thickness and drop time deviation from free fall. In addition, the plummet rebound height, which is an indication of the rigidity of the tester and foundation, appears to require some measure of control; although the effect of this variable on the reported threshold values was not conclusive.

LIST OF REFERENCES

1. Beane, G. A., "Results on Lox-Lubricant Impact Sensitivity Cooperative Test Program No. 1," WADC Technical Note 58-344, November 1958.

2. Letter from Marc P. Dunnam, ASD, Subject: Initiation of Cooperative Program No. 2, March 4, 1960.

3. Letter from G. A. Beane, ASD, Subject: Result of LOX-Lubricant Impact Sensitivity Cooperative Testing Program No. 2, November 8, 1960.

4. Baber, B. B., et al., "Lubrication Research and Test Method Development for Aerospace Propulsion Systems (March 15, 1960 to January 14, 1961)," ASD Technical Report 61-85, May 1961.

APPENDIX I

USAF SPECIFICATION BULLETIN 527

LIQUID OXYGEN COMPATIBILITY IMPACT SENSITIVITY TEST METHOD

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Figures

1. Scope and purpose.- This bulletin covers a test method for determining the compatibility of materials with liquid oxygen using the ABMA type impact tester. The materials included in the scope of this procedure are liquids (oils), greases, solids, solvents, and coatings (dry film).

2. Brief outline of the impact sensitivity test method.- Samples of the material are prepared in standard impact-test specimen cups, the material precooled and the cups filled with liquid oxygen and placed in an impact tester. A precooled striker pin is centered in the cup. A plummet is released from a given height on the striker pin, which in turn impacts the material. Observation for reaction is made and the impact sensitivity determined.

3. Test equipment

3.1 Anvil region assembly.- The anvil region assembly (a critical portion of the ABMA type impact tester) shown on figure 1 shall be used in all testing conducted in accordance with this bulletin. (See 8.4.)

3.2 ABMA type impact tester.- The ABMA type impact tester (see figure 2, 8.3, and 8.4) is the standard test equipment for determining the compatibility of a material with liquid oxygen. The complete tester should be used in testing whenever possible. It consists of three guide tracks capable of maintaining vertical alignment under repeated shock conditions, plummet weight, electromagnet for supporting or releasing the plummet, solenoid operated safety catch to support the plummet when the magnet is not energized, base plate supported by reinforced concrete, anvil plate, striker pin, striker pin guide, specimen cup holder, and specimen cup.

3.2.1 Magnet.- The magnet is designed to hold over 20 pounds of weight with a minimum amount of electrical energy. The voltage required is 6 volts d.c.

3.2.2 Safety catch.- The safety catch is designed to hold the plummet near the base of the magnet. It is used to support the plummet during the cleaning operation when the magnet is not energized.

3.2.3 Base plate.- The base plate of 1-inch thick stainless steel rests on a 1/8-inch thick stainless steel sheet. This sheet covers the 2-foot cube base of reinforced concrete to prevent scattering of cement dust upon impact. Four stainless steel foundation bolts and nuts protruding from the concrete base are used to fasten the plate and sheet to the concrete.

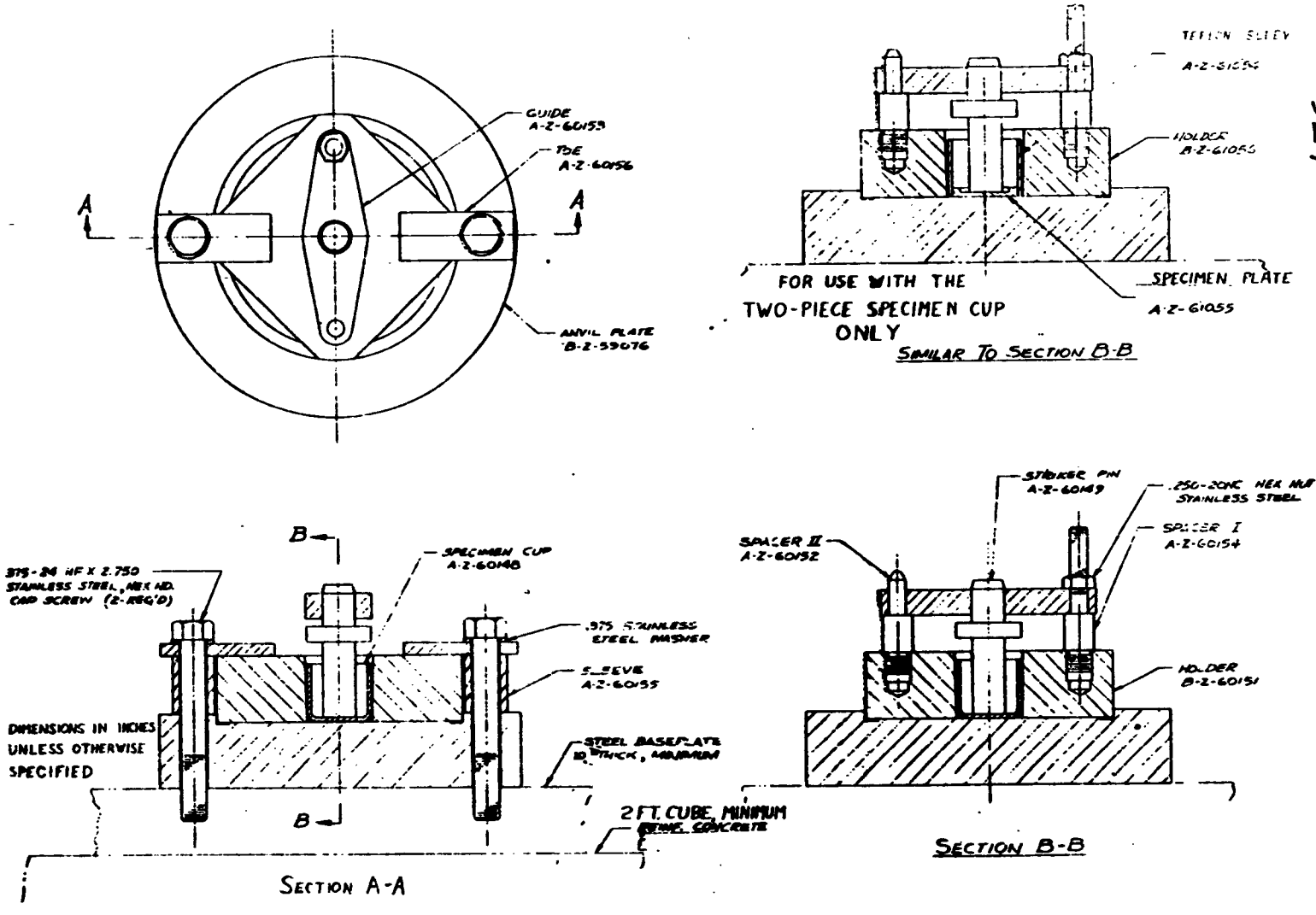


FIGURE 1. ANVIL REGION ASSEMBLY (DWG NR. C-Z-61006)

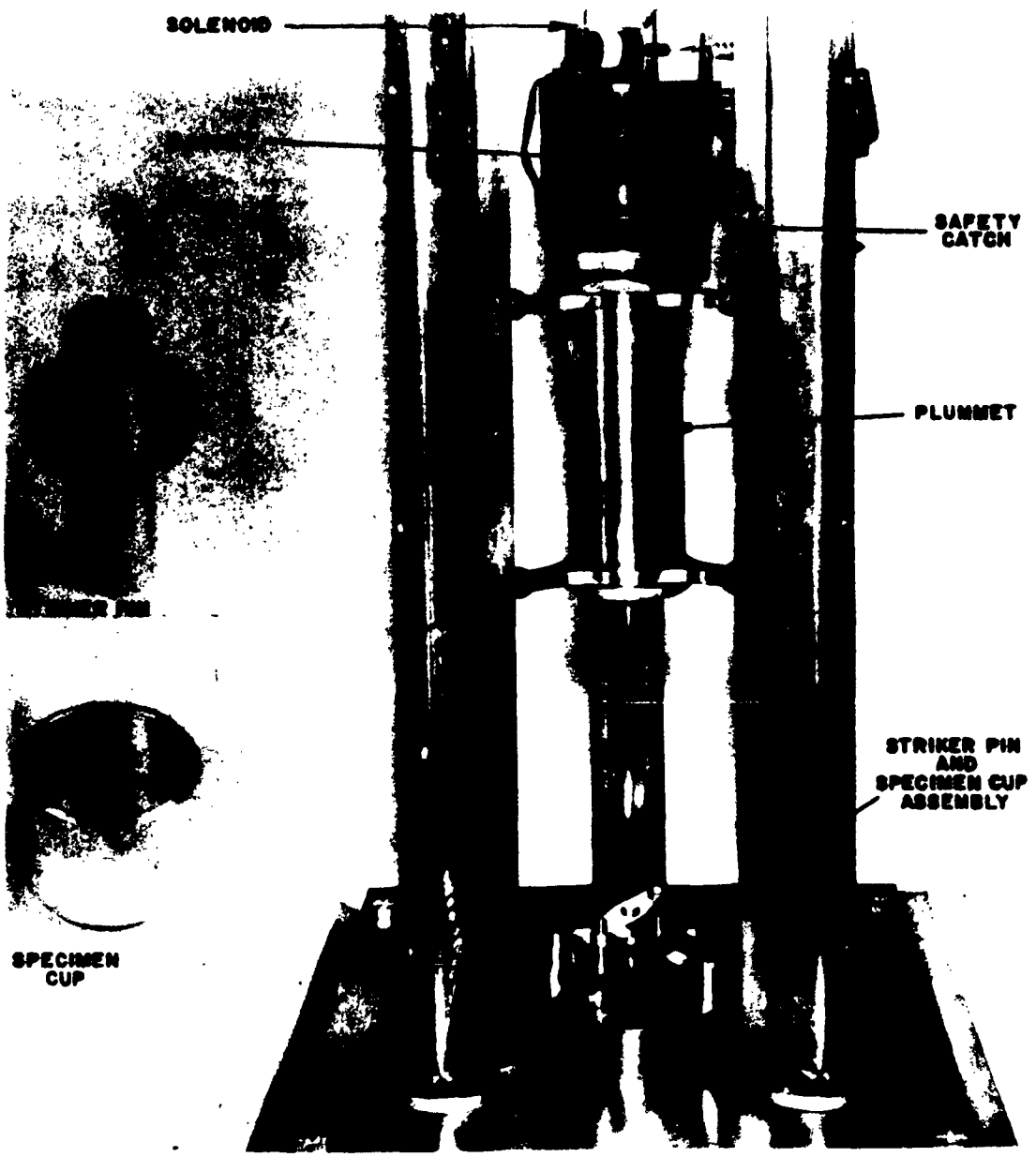


FIGURE 2.
PHOTOGRAPH OF ABMA IMPACT TESTER
STRIKER PIN AND SPECIMEN CUP

3.2.4 Anvil plate.- The anvil plate is an approximately 1-inch thick heat-treated stainless steel plate that is centered and rests on the base plate. This plate centers the specimen cup holder and provides protection from denting the base plate upon impact.

3.2.5 Striker pins.- The striker pins are made from 17-4 PH stainless steel. Sufficient pins should be provided for testing and discard. (See 8.1.)

3.2.6 Specimen cup holder.- The specimen cup holder is a 1-inch thick stainless steel block and is centered on the anvil plate by a pilot circle machined in the anvil plate. The specimen cup holder contains two protruding spacers which align the striker pin guide, and in turn the striker pin with the nose of the plummet, thus ensuring a direct hit by the nose of the plummet on the striker pin in the specimen cup.

3.2.7 Specimen cups.- One-piece and two-piece specimen cups are used in this test method. A one-piece aluminum specimen cup shall be used in the testing of liquids (oils), solids, and solvents. A two-piece specimen cup consisting of an aluminum specimen plate and teflon sleeve shall be used in the testing of greases and coatings (dry film). (See 8.2 and 8.6.)

3.3 Auxiliary equipment.- The auxiliary equipment consists of forceps for handling the specimen cups and striker pins, stainless steel spatulas, liquid oxygen handling equipment such as stainless steel Dewar flasks, liquid oxygen protective gloves, lintless laboratory coat, safety goggles, gaseous oxygen bottles, and liquid oxygen storage containers. Additional handling equipment includes striker pin holders (see figure 3), specimen cup trays, specimen plate tee and positioner (see figure 4), desiccators (without desiccant) for storing specimen cups and striker pins, a 10 milliliter (ml) microburette, a stainless steal top (level) work table, a test cell for the apparatus with a protective window for observation of test drops, a control panel for the operator to activate the safety catch and electromagnet, and timing instrumentation to measure the drop time of the plummet (see figure 5).

3.3.1 Forceps.- Stainless steel tongs or forceps shall be used to handle the cleaned specimen cups, striker pins, and solid samples to prevent contamination by the operator's hands.

3.3.2 Liquid oxygen storage containers.- Liquid oxygen storage containers are required for storage of the liquid oxygen.

3.3.3 Gaseous oxygen bottles.- Gaseous oxygen bottles should be used to transfer liquid oxygen from the storage containers to the Dewar flasks.

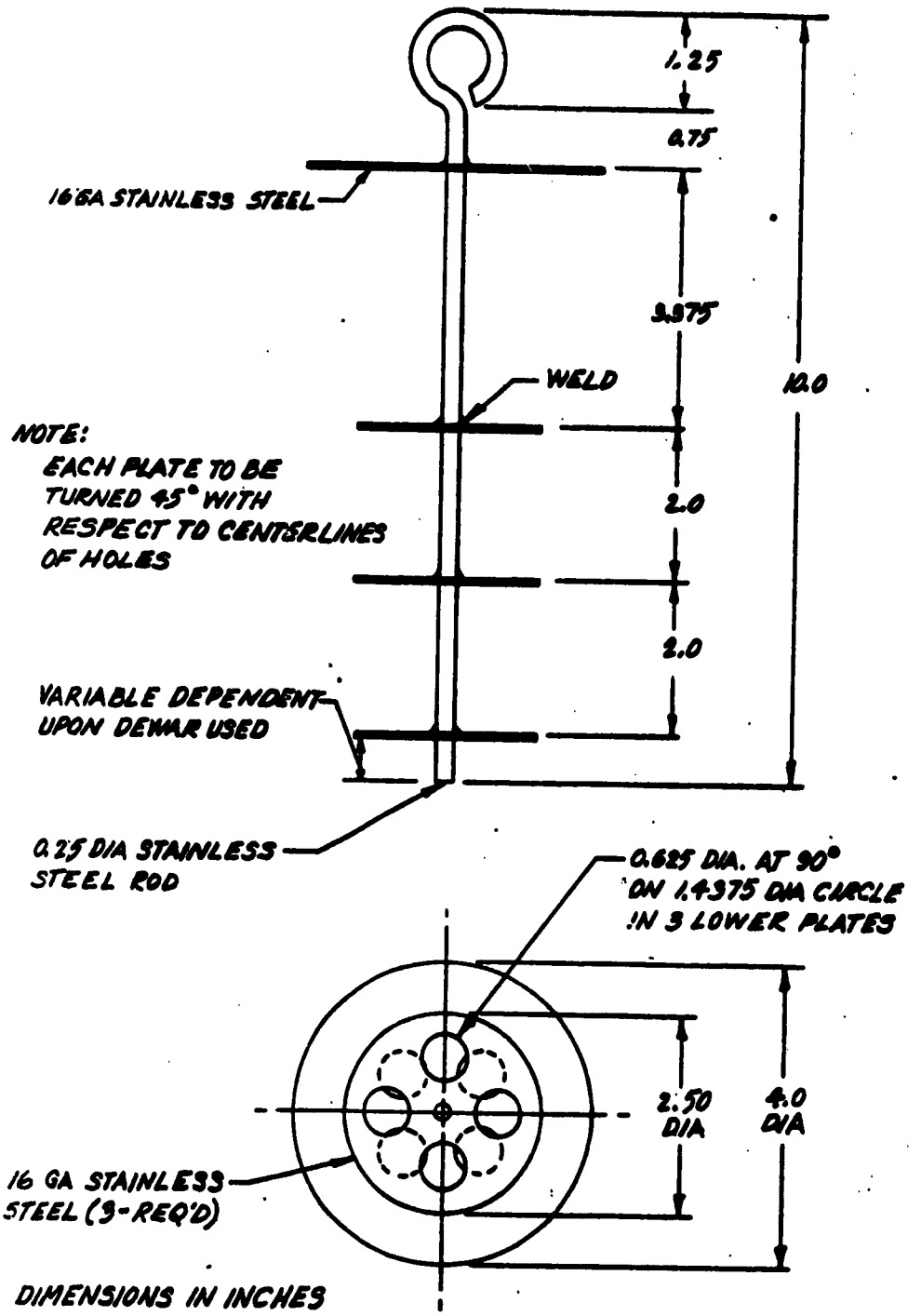
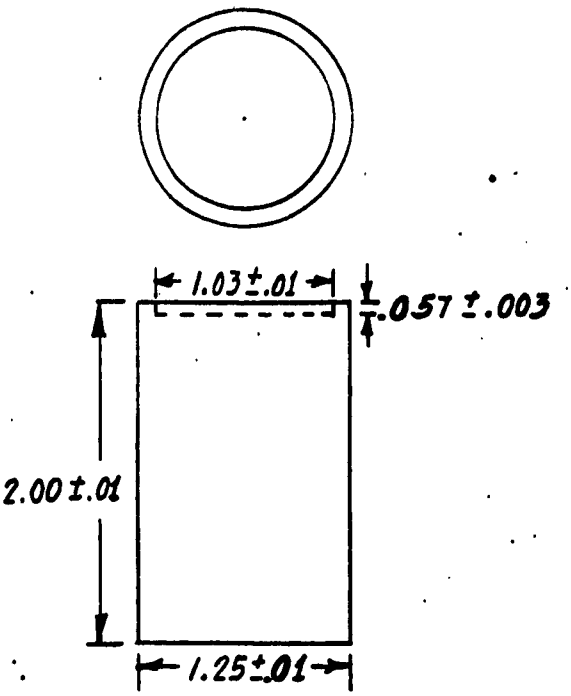
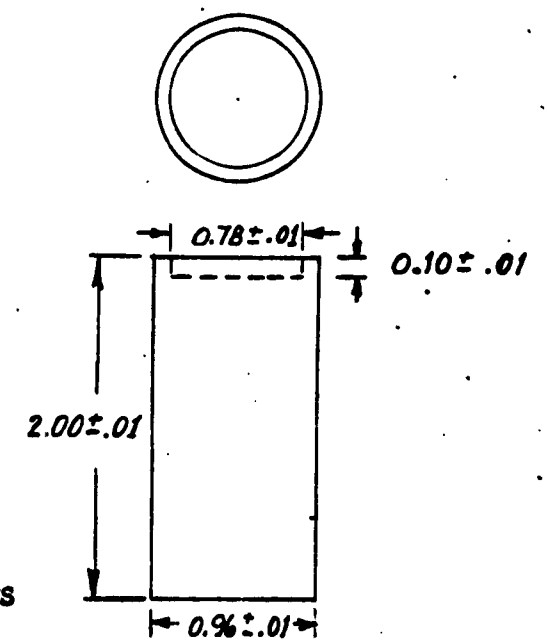


FIGURE 3. DRAWING OF STRIKER PIN HOLDER



Specimen Plate Tee



DIMENSIONS IN INCHES

Specimen Plate Positioner

FIGURE 4. SPECIMEN PLATE TEE & POSITIONER

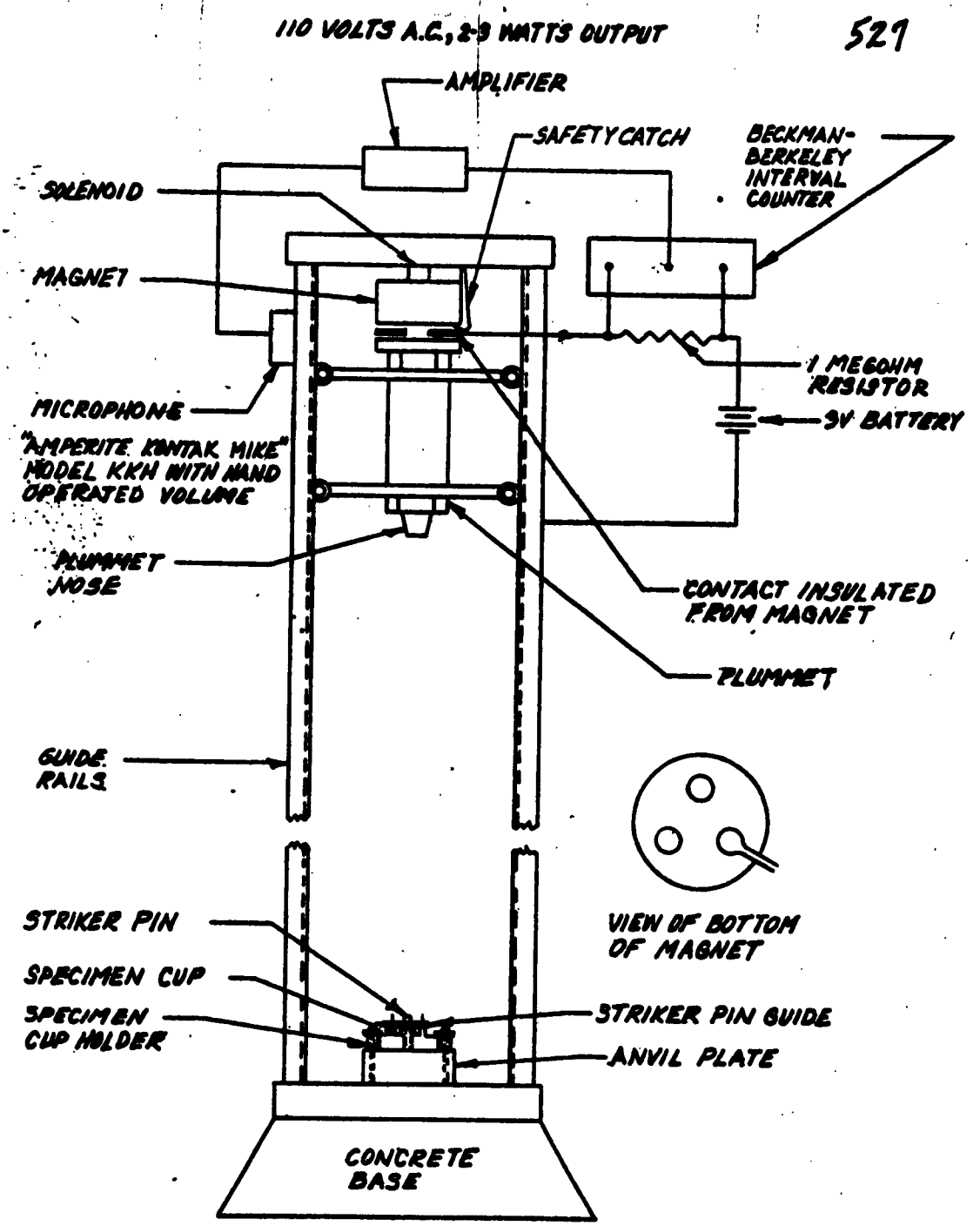


FIGURE 5. INSTRUMENTATION FOR MEASUREMENT OF DROP TIME

3.3.4 Test cell.- The impact tester shall be housed in a test cell containing a concrete floor. Aluminum sheeting shall be used to cover the ceiling of the cell, and, if possible, the walls. This facilitates cleanliness. The cell shall contain an observation window of Plexiglas, or other shatter-proof material. It shall be shrouded to darken sufficiently for observation of flashes. Continuous ventilation shall provide fresh, uncontaminated, filtered air to the test cell.

3.3.5 Timing instrumentation.- A Berkeley Universal Counter and Timer, Model 5500, or equivalent, shall be used for measurement of drop time (see figure 5 and 8.5). Two contacts are attached to the tester to start and stop the counter. The overall drop time is measured from any given height of the impact tester. The timing instrument is necessary to permit evaluation of the kinetic energy expended in friction.

4. Materials

4.1 Liquid oxygen.- Liquid oxygen specified in this bulletin shall conform to Specification MIL-O-25508.

4.2 Trichloroethylene.- Extraction grade trichloroethylene specified in this bulletin shall have a maximum nonvolatile residue content of 0.0010 percent by weight.

4.3 Alkaline cleaner.- The formula for the alkaline cleaner specified in this bulletin shall be: 15 grams trisodium phosphate (Na_3PO_4) and 15 grams sodium hydroxide (NaOH) in 1 liter of distilled water.

5. Cleaning and storage of equipment

5.1 General.- The impact tester, its accessories, and the test cell shall be maintained in a clean condition. The guide tracks, plummet, anvil plate, striker pin guide, specimen cup holder, and base plate of the impact tester shall be cleaned thoroughly at the start of each test and between tests of different materials by using steel wool, and rinsing with clean extraction grade trichloroethylene (see 4.2). In addition, the anvil plate, specimen cup holder, striker pin guide, and plummet nose shall be cleaned with clean extraction grade trichloroethylene at least after impact test of every tenth sample during test. After completion of testing for the day, the impact tester, handling equipment (Dewar flasks and forceps), striker pins used in testing, and sample preparation equipment shall be rinsed with clean extraction grade trichloroethylene. Cleanliness must be maintained throughout any series of impact testing to minimize erroneous test results.

5.2 Test cell.- The aluminum ceiling and any fixtures in the test cell that could accumulate dust shall be cleaned weekly. The concrete floor shall be vacuumed weekly and scrubbed when necessary. The air intake shall be filtered and the filter changed at least every 3 months.

5.3 Striker pins.- Striker pins shall be cleaned as follows: Vapor degrease with trichloroethylene, follow with a 15-minute soak in alkaline cleaner (see 4.3), rinse thoroughly with distilled water, and nitrogen (water pumped only) or oven dry. (See 3.3.1.) Visually examine pins for discoloration, and scratches or pits on the striking area. Discolored pins may be recleaned, failing this, they should be remachined (see 5.3.2) and recleaned, or discarded. Pins containing scratches or pits shall be remachined and recleaned, or discarded.

5.3.1 Storage of cleaned striker pins.- Cleaned striker pins shall be stored in a clean desiccator (without desiccant) until use. The cleaning specified in 5.3, except for the vapor degrease, shall be repeated on pins stored 1 month or more. Pins stored more than 6 days but less than 1 month shall be given a rinse with clean extraction grade trichloroethylene and nitrogen (water pumped only) or oven dried prior to use.

5.3.2 Remachined striker pins.- The length of remachined striker pins shall be 2.000 plus 0.010 or minus 0.100 inches. Striker pins shall be remachined if the striking area contains scratches or pits, or both. It may be necessary to remachine pins bent or flattened during impact test. Badly bent pins and remachined pins in which the diameter near the striking area exceeds 0.505 inch or is less than 0.495 inch shall be discarded.

5.4 Specimen cups.- Specimen cups shall be cleaned as follows: Vapor degrease with trichloroethylene, wash with detergent (Tide, or equivalent), rinse with distilled water, and nitrogen (water pumped only) or oven dry. (See 3.3.1.) Visually examine one-piece cups and specimen plates (aluminum portion) of two-piece cups for discoloration and scratches or pits of the interior base. Discolored cups and specimen plates may be recleaned, failing this, discard as well as those containing scratches or pits. Specimen cups and specimen plates shall be used only once and discarded. Visually examine teflon sleeves of two-piece cups for cleanliness. Unclean sleeves may be recleaned, failing this, discard. Teflon sleeves may be reused until no longer cleanable or no longer a proper fit, or both.

5.4.1 Storage of cleaned specimen cups.- Cleaned specimen cups shall be stored in a clean desiccator (without desiccant) until use. The cleaning specified in 5.4, except for vapor degrease, shall be repeated on cups stored 1 month or more. Cups stored more than

6 days but less than 1 month shall be given a rinse with clean extraction grade trichloroethylene and nitrogen (water pumped only) or oven dried prior to use.

5.5 Auxiliary equipment

5.5.1 Stainless steel ware.- Stainless steel ware, such as striker pin holders, forceps, spatulas, and specimen cup trays, shall be cleaned in the same manner as the striker pins (see 5.3). Once integrated into the handling procedure, a thorough rinse of the stainless steel ware with clean extraction grade trichloroethylene is the only cleaning necessary.

5.5.2 Glassware.- Any glassware, such as microburette, beakers, and pipette shall be cleaned as follows: Soak approximately 2 hours (depending on degree of contamination) in warm sulfuric acid-dichromate solution, rinse with distilled water, and nitrogen (water pumped only) or oven dry.

5.5.3 Liquid oxygen handling equipment.- Liquid oxygen handling equipment, such as gaseous oxygen lines (tubing), and liquid oxygen storage containers shall be cleaned thoroughly at least every 3 months with clean extraction grade trichloroethylene. Safety manuals on liquid oxygen handling and cleaning procedures shall be followed.

5.6 Cleanliness check.- The effectiveness of the cleaning procedure described herein shall be checked as follows: Select at least five each cleaned specimen cups and striker pins from every batch cleaned. Precool the blank (empty) specimen cups, striker pins, and specimen cup holder as specified in 6.2.3.1, 6.2.4, and 6.2.5, respectively. From a height of 48 inches perform test drop as specified in 6.3.3. If no reaction occurs, the specimen cups, striker pins, and test rig are considered clean. Should a reaction occur, the cups and striker pins represented by those tested, and the test rig shall be recleaned and the check procedure herein repeated.

6. Testing

6.1 Preparation of test samples

6.1.1 Number of samples.- At least 20 samples but not more than an amount that can be tested within 8 hours after preparation should be prepared in accordance with 6.1.2 through 6.1.6, as applicable, for each series of test drops. More than 20 samples may be desirable to allow for rejection due to samples floating or breaking up during precooling (see 6.2).

6.1.2 Liquids (oils).- Liquid samples shall be prepared as follows: Shake liquid well before use and allow air bubbles to float out of solution. Utilize a 10 ml microburette in handling the liquids.

Determine by trial and error technique the volume of liquid required to give a sample thickness of 0.050 inch in the specimen cup. (This is necessary because of variations in density and surface tension from liquid to liquid.) Fill the cleaned, one-piece specimen cup to a sample thickness of 0.050 inch. A micrometer depth gage with leveling blocks is suggested for measurement and the work table should be level. Proceed as specified in 6.2.3.2.

6.1.3 Greases.- Grease samples shall be prepared as follows: Place the cleaned specimen plate of the two-piece cup in the cleaned specimen plate tee. Press sufficient sample material (a slight excess) in the specimen plate with the cleaned stainless steel spatula to form a uniform sample free of air bubbles and void spots. Scrape off the excess sample grease with the spatula until a smooth surface level with the edge of the specimen plate is achieved. Remove excess sample from the outer perimeter of the specimen plate with clean lintless tissue. Assemble the two-piece specimen cup as described in 6.1.3.1 and proceed as specified in 6.2.3.2.

6.1.3.1 Assemblage of the two-piece specimen cup.- Assemble the two-piece specimen cup as follows: Place the specimen plate on a clean, flat surface. Carefully press the teflon sleeve down around the specimen plate. A specimen plate positioner (see figure 5) may be used to hold the specimen plate during this procedure.

6.1.4 Solids.- Samples of solids from the application products shall be cut and prepared as follows: The diameter of the sample should be small enough to fit within the flat portion of the bottom of the specimen cup and greater than the diameter of the striker pin. When the application product has a thickness less than 0.050 inch and can be cut to form a reasonably rigid disc (not too flimsy) with a diameter of 0.750 inch (tolerance of minus 0.125 inch), test samples shall be prepared therefrom. When the application product has a thickness greater than 0.050 inch or cannot be cut to form a reasonably rigid disc (not too flimsy) having the required diameter, flat-disc test samples 0.050 inch ±0.003 inch thick and 0.750 inch (tolerance of minus 0.125 inch) diameter shall be specially prepared. These discs shall be made from the same identifiable lot of sheet or raw material from which the application product was made. The surface finish of both sides of the discs shall assimilate the application product. Measure thickness of each sample specimen with a micrometer, and record same. Clean sample specimen by thoroughly rinsing in clean extraction grade trichloroethylene, and nitrogen (water pumped only) or oven dry. (See 3.3.1.) Should the trichloroethylene have an adverse effect on the sample, use any other liquid oxygen compatible solvent that has no reaction out of 20 test drops at 70 ft lb when subjected to procedure specified in 6.1.5.1 or 6.1.5.2. Should all such compatible solvents have an adverse effect on the sample disc, clean with a detergent wash (Tide or equivalent), rinse with distilled water, and nitrogen (water pumped only) or oven dry. Place in cleaned one-piece specimen cup, and proceed in accordance with 6.2.3.2.

6.1.5 Solvents.- Solvent samples shall be prepared as specified in 6.1.5.1 or 6.1.5.2.

6.1.5.1 Method I.- Concentrate the solvent to 2 percent of its original volume by evaporating the solvent in a gravity convection explosion-proof oven. Hold the temperature of the oven at 5° to 10° F below the boiling point of the evaporating solvent. Place the 2 percent concentrated solvent in the cleaned, one-piece specimen cup to a thickness of 0.050 inch ±0.003 inch. Proceed as specified in 6.2.3.2.

6.1.5.2 Method II.- Place 5 cc of the 2 percent concentrated solvent (see 6.1.5.1) in the cleaned, one-piece specimen cup and further evaporate as described in 6.1.5.1, until dry. Proceed as specified in 6.2.3.2. The sample thickness of this residue obviously will be less than 0.050 inch.

6.1.6 Coatings.- Coating samples such as dry film lubricants and paints shall be prepared as follows: Apply the coating to the inside of the cleaned specimen plate of the two-piece cup in the same manner and to the same thickness that is intended for its hardware application; e.g., sprayed on, baked on, brushed on, et cetera. Upon completion of the drying or baking process, assemble the two-piece specimen cup as described in 6.1.3.1. Proceed as specified in 6.2.3.2. The sample thickness of this coating probably will be less than 0.050 inch.

6.2 Precooling

6.2.1 Definition.- The term precooling as used herein means to lower the temperature of the material being cooled to the boiling point of liquid oxygen at one atmospheric pressure (-297° F).

6.2.2 Dewar flasks.- Dewar flasks shall be precooled as follows: Transfer a small volume of liquid oxygen from the storage container into the Dewar flask and cool flask to liquid oxygen temperature. (Liquid oxygen boils vigorously until the surface of the Dewar flask has cooled to liquid oxygen temperature.) When cooled, fill flask to three-fourths of its total volume with liquid oxygen. Cover with clean aluminum foil or stainless steel cover. (Experience has established that the economical method is to use stainless steel wide-mouthed Dewar flasks and stainless steel covers.)

6.2.3 Specimen cups

6.2.3.1 Blank specimen cups.- The cleaned, blank specimen cups shall be precooled by slow immersion in a Dewar flask containing liquid oxygen.

6.2.3.2 Specimen cups containing sample material.- The specimen cups containing sample material to be tested (see 6.1) shall be precooled as follows: Proceed with extreme care to minimize cracking of the sample in the base of the cup. On a level table, place a stainless steel tray (similar to a hospital surgical pan) and put specimen cups containing sample material therein. (The use of the level table assures a uniform sample thickness.) Slowly pour liquid oxygen into the tray to a level one-fourth the height of the specimen cups. Avoid splashing small bubbles of boiling liquid oxygen onto the warm test sample in order to prevent a hole or void spot in the sample. After the sample material has frozen, pour sufficient liquid oxygen into the tray to float the cups. (Do not pour liquid oxygen directly into the cups as this will increase the danger of breakup of the sample and may cause the sample to pop from the cup.) Slowly submerge the cups by allowing the liquid oxygen to overflow the sides of the cup. Make a visual check to assure sample does not contain a void or hole, and that the material has not separated from the bottom of the cup, or popped out. Discard any sample cup that does not pass this inspection. Samples need not be discarded for cracking. Perform impact test (see 6.3) within 4 hours after precool.

6.2.4 Striker pins.- Cleaned striker pins shall be precooled by placing pins in striker pin holder and immersing in a Dewar flask containing liquid oxygen.

6.2.5 Specimen cup holder and anvil plate.- The specimen cup holder and anvil plate (anvil region) shall be precooled just prior to placing a specimen cup for test, as follows: Use cleaned forceps and specimen cup or special transfer container to transfer liquid oxygen from the Dewar flask to the anvil region. Pour liquid oxygen into the hole of the specimen cup holder and onto the anvil plate until boiling of the liquid oxygen is negligible. This precooling allows sufficient time for the operator to leave the test cell and impact the sample while it is in contact with the liquid oxygen.

6.3 Impact test

6.3.1 Critical test parameters.- Critical test parameters are as follows:

a. Plummet weight.- Plummet weight shall be 20 ±0.05 pounds.

b. Drop height.- Drop height shall not deviate more than ±0.2 inch from the specified height.

c. Striker pin diameter.- Striker pin diameter near the striking area shall be 0.500 ±0.005 inch.

d. Sample thickness.- Sample thickness shall be 0.050 ±0.003 inch, and as otherwise specified in 6.1.4, 6.1.5, and 6.1.6.

e. Energy loss.- The energy loss due to friction shall be indirectly controlled by measuring the overall drop time for each drop from any given drop height. The average measureddrop time for 20 valid test drops from the same drop height shall not exceed the computed theoretical free fall time by more than 3 percent. An individual test drop is considered valid when its measured drop time does not deviate by more than 0.010 second from the average measured drop time of 20 test drops at the same drop height. An individual test drop is also considered valid when a reaction occurs regardless of its measured drop time.

6.3.2 Blank drop.- To determine cleanliness, at least two blank drops shall be made from a height of 48 inches prior to the first and one after every tenth drop of each sample material in accordance with 6.3.3. If no reaction occurs, testing of sample material may proceed. Should a reaction occur, the general cleaning specified in 5.1 shall be repeated and five blank drops performed thereafter. Should a reaction still occur, the entire cleaning procedure specified in section 5 shall be repeated until five blank drops are made without reaction.

6.3.3 Test drop.- The following steps shall be accomplished with sufficient care and speed so that the specimen cup will contain some liquid oxygen at all times prior to impact: Adjust magnet to proper drop height. Use clean forceps to set the precooled specimen cup (blank or containing the frozen sample and liquid oxygen, as applicable) into the precooled cup holder of the anvil region assembly. Make a final visual check to assure the frozen sample has not separated from the bottom of the cup. Discard those that have separated. Center the precooled striker pin into the cup and hold in position by the striker pin guide. Replenish the partially boiled-away liquid oxygen by topping the cup with liquid oxygen. Cover the exposed containers of liquid oxygen (liquid oxygen tray containing sample cups and Dewar flask containing striker pins and pin holder). Close test cell door, turn off cell lights, and set timer. Release safety catch and plummet by means of the electrical control panel located outside the test cell near the observation window. Observe and record the results of the impact reaction as defined in 6.3.4. Record drop time of the plummet.

6.3.4 Impact reaction.- A positive reaction is considered to be a detonation, visible flash during impact, or discoloration, burnt or charred spots, pits, or any such evidence of a reaction upon post

inspection of the rewarmed striker pin and the sample specimen cup. Odor alone will not be considered to be a reaction. Degree of severity of reaction shall be noted as follows:

Extreme - Obvious cup deformation due to reaction.

Moderate - Obvious flash, audible detonation, burning, or any combination thereof.

Faint - Barely detectable flash or detonation, or combination thereof.

Char - Evidence of burnt or charred spots on either the striker pin or specimen cup.

If any reaction occurs on rebound, it should be reported and classified in the same manner.

6.3.5 Interpretation of results.- Unless otherwise specified in the procurement specification, when no reaction in a minimum of 20 consecutive drops or a maximum of 1 reaction in 40 consecutive drops occurs, the material shall be considered to pass this test.

7. Report.- The following data should be reported for each series of 20 test drops:

a. Type of sample material tested, its commercial trade name, and batch number.

b. Drop height of the plummet.

c. Measured free fall time of the plummet for each test drop.

d. Computed theoretical free-fall drop time for each drop height.

e. Percent of deviation of average measured drop time from theoretical free-fall drop time.

f. Ambient temperature, humidity, and barometric pressure, gravitational constant.

g. Sample thickness.

h. Number and severity of reactions that occurred.

i. Number of blank cups tested.

j. Date, testing laboratory, and the name of operator conducting test.

8. Notes

8.1 Known sources of supply for the expendable striker pins (see 3.2.5) are: (1) D. S. Anthony and Sons, 1235 W. Laurel Street, San Antonio, Texas, and (2) Dobbins Metal Company, 1207 Fulton Avenue, San Antonio, Texas.

8.2 A known source of supply for the expendable specimen cups (see 3.2.7) is Dobbins Metal Company, 1207 Fulton Avenue, San Antonio, Texas.

8.3 Know sources of supply for the ABMA type impact tester (see 3.2) are: (1) Gray Schultz Inc., Mt. Elliott Avenue, Detroit 34, Michigan, and (2) Peerless Engineering Company, Inc., 4741 Firestone Boulevard, South Gate, California.

8.4 Complete sets of drawings for the ABMA type impact tester and the anvil region assembly will be furnished upon written request to: Aeronautical Systems Division, Attn: ASRCEF-2, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio.

8.5 A known source of supply for the Berkeley Universal Counter and Timer, Model 5500 (see 3.3.5) is Berkeley Scientific Division of Beckman Instruments, Inc., Richmond, California.

8.6 A known source of supply for teflon tubing for the teflon sleeve (see 3.7) is the Sparta Manufacturing Company, Dover, Ohio.

8.7 Determination of threshold value

8.7.1 Approximate threshold value.- The approximate threshold value should be sought in accordance with the schedule of drop heights listed in table I. Testing at each drop height should continue until either 20 no-reaction drops or 1 reaction drop occurs. The approximate threshold value is the potential energy level for the first height at which no reaction occurred in 20 drops.

Table I. Schedule of drop heights

Schedule	Drop height (inches)
1st drop series	42
2d drop series	33
3d drop series	24
4th drop series	15

8.7.2 Definitive threshold value.- The definitive threshold value should be sought at consecutively decreasing drop heights shown in

table II beginning two drop heights (6 inches) above the height at which the approximate threshold value (see 8.7.1) was accomplished. Testing at each drop height should continue until either 20 no-reaction drops or 1 reaction drop occurs. The definitive threshold value is the potential energy level for the higher of the two highest adjacent drop heights at which no reaction occurred in 20 drops, and below which level no reaction occurred.

Table II. Drop height and potential energy level

Drop height (inches)	Potential energy (ft lb)	Drop height (inches)	Potential energy (ft lb)
48	80	27	45
45	75	24	40
42	70	21	35
39	65	18	30
36	60	15	25
33	55	12	20
30	50	9	15

APPENDIX II

PRECOOLING PROCEDURE USED FOR COOPERATIVE TEST PROGRAM NO. 3

PRECOOLING PROCEDURE

TO REPLACE PARAGRAPH 6.2.3.2 OF SPECIFICATION BULLETIN 527

(For Co-op Program No. 3 Only)

6.2.3.2 Specimen cups containing sample material. - The specimen cups containing sample material to be tested (see 6.1) shall be precooled as follows: Proceed with extreme care to minimize cracking of the sample in the base of the cup. On a level table, place an insulated stainless steel tray (1) and place specimen cups containing sample material therein. (The use of the level table assures a uniform sample thickness.) Liquid oxygen is then poured into the tray by means of a small mouth transfer container (2) and a filling funnel. (3) The funnel is fitted with a metal plug containing a 1/16 in. orifice (4) which controls the rate at which the liquid oxygen is added. The funnel is held in position with the tip of the metal plug approximately 1/4 in. from the bottom of the tray. Liquid oxygen is then added to the funnel to maintain a steady flow through the orifice until all of the samples are frozen. After the test samples are frozen, the funnel is then removed and liquid oxygen added directly to the tray from the transfer container until there is a sufficient quantity of liquid oxygen in the tray to float the specimen cups. The specimen cups are then submerged by slowly tilting the cups and allowing the liquid oxygen to overflow the sides of the cup. Make a visual check of all the frozen samples to assure that the samples do not contain a void or hole, and that the sample material has not separated from the bottom of the cup, or popped out. Discard any test sample that does not pass this inspection. Samples need not be discarded for cracking. Perform impact test (see 6.3) within 4 hours after precool.

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(1) "Vollrath" stainless steel pan with cover or equivalent. Dimensions: 12 in. x 8 in. x 2 in. Approximate cost - $11.00. Insulation is provided by asbestos tape or equivalent.

(2) "Purox" type C laboratory container or equivalent. Capacity: 1 liter. Manufactured by Linde Company. Approximate cost - $26.00.

(3) Filling funnel for "Purox" type containers. Manufactured by Linde Company. Approximate cost - $5.00

(4) Sketch of plug shown in attached Figure 1.

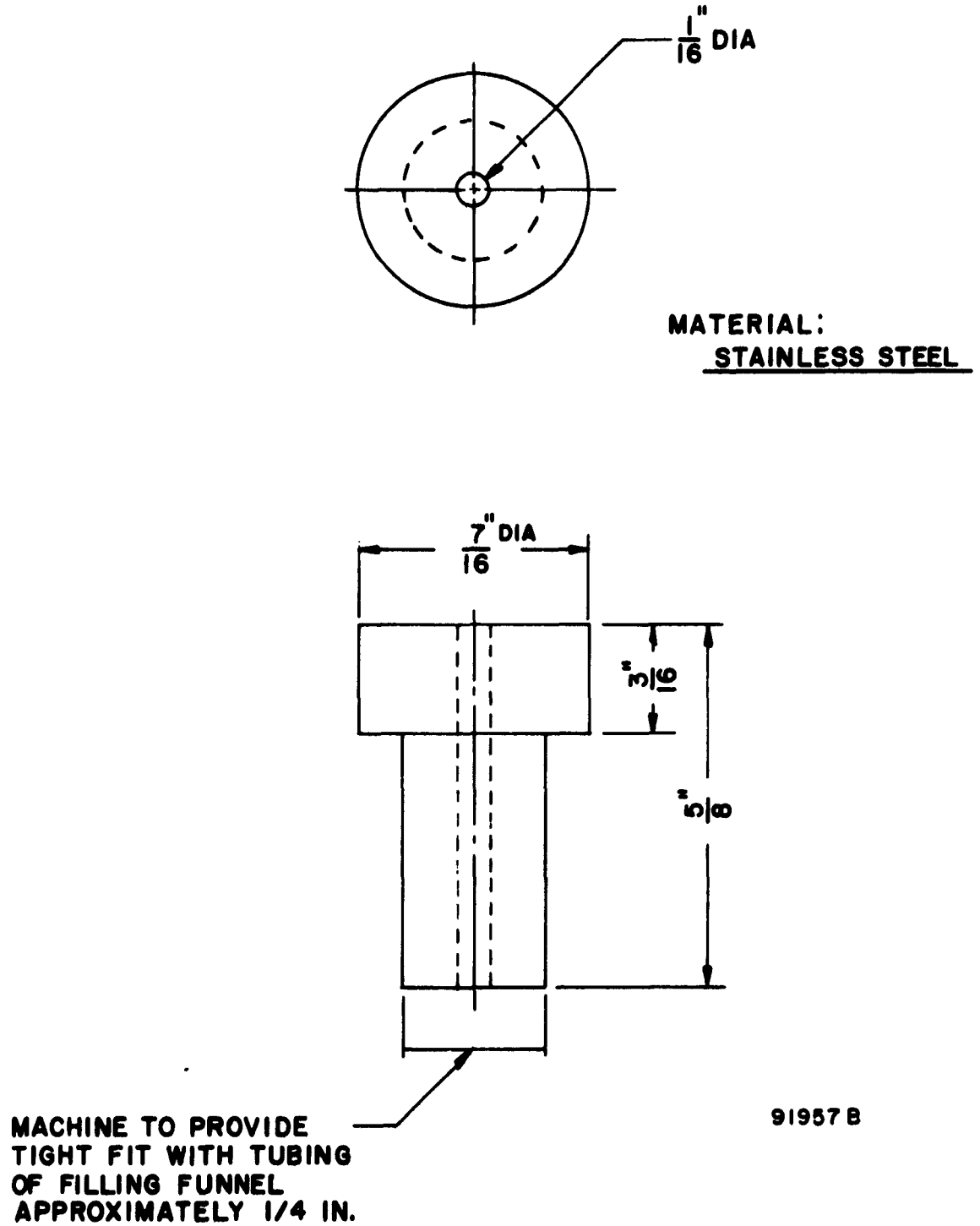


FIGURE 1. METAL PLUG FOR FILLING FUNNEL

APPENDIX III

RMD IMPACT TESTER OPERATING INSTRUCTIONS

(Reproduced from Report No. CMP 91, Reaction Motors Division, Thiokol Chemical Corporation)

I. DESCRIPTION

The Reaction Motors Impact Tester is a convenient laboratory tool for determining the shock sensitivity of various materials in the presence of liquefied gases, liquids, or gases. The impact test is a simple means of quantitative determination of the relative sensitivity of materials to initiation of an explosion. It is not a measure of a fundamental property. Your impact tester has been determined, on the basis of exhaustive tests, to provide the most efficient and reliable method of obtaining accurate and reproducible shock sensitivity data for various propellants as well as for lubricants, sealants, plastic and elastomeric materials in the presence of cryogenic or other oxidizing media.

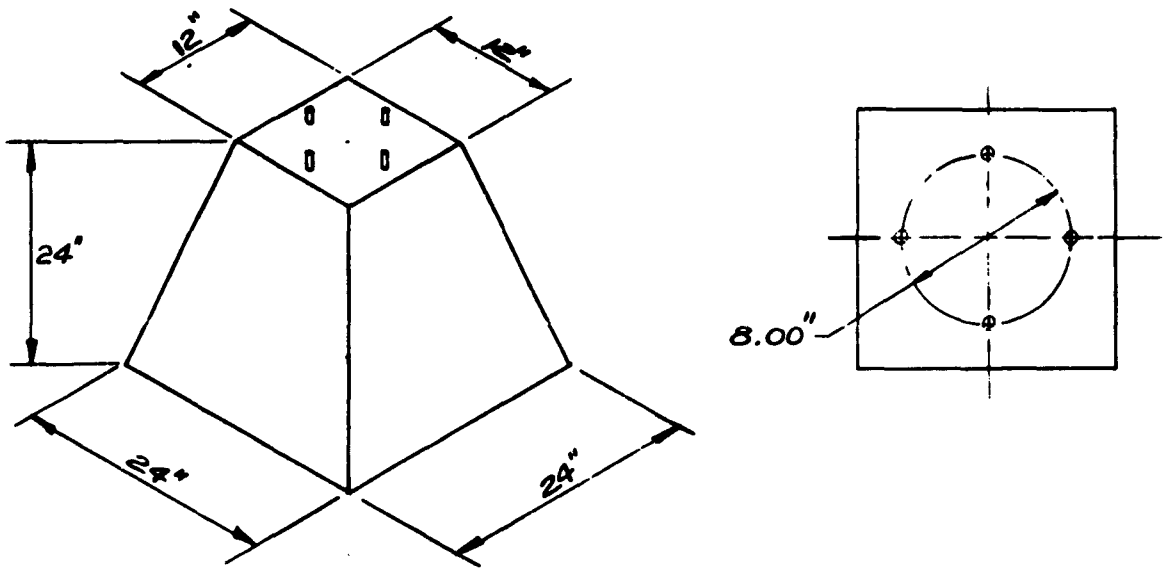
Your Impact Tester (reference Figure 1) is delivered completely assembled. You need only mount the unit on a concrete pad, plug it in to a 110 volt AC 60 cycle supply and follow the simple instructions and procedures described herein to obtain accurate shock sensitivity test data. It consists of a platform base and die cup support, a guide frame comprising three precision ground rods, a top support plate, two plummet assemblies of different weights to permit a greater range of impact force, and an electrically controlled plummet release platform. Also included is a representative sampling of expendable items including 100 each of two different die cup assemblies for impact testing with different materials and surrounding media and fifty plastic containers for testing with cryogenic fluids.

The plummet release platform is free to travel vertically and is secured at the selected height by a locking knob. Raising or lowering the platform provides for plummet release from various heights above the test sample, thus permitting the impact energy of the plummet to be varied in small increments. A scale extending between the base and the top support plate is positioned so that the pointer on the plummet platform shows the height in inches of the plummet striker, above the test sample. A momentary "on" push button switch is provided which, when depressed, actuates the solenoid on the plummet release platform. The energized solenoid coil withdraws the spring loaded latch thus permitting the plummet to fall freely. Friction between the plummet and the guide rods is minimized by low friction rings on the plummet bearing surface.

A transparent plexiglass shield is provided and should be placed around the base of the impact tester so that small fragments released during an impact explosion will be restricted to the impact area.

II. INSTALLATION PROCEDURE

The Impact Tester as delivered is completely assembled. It should be mounted on a concrete pad to assure minimum deflections and energy absorption of the mounting provisions. A typical concrete mounting pad is shows below:



A comfortable working height for the tester can be obtained by providing a mounting pad height of from 24 to 30 inches. Four 1/2-inch diameter bolts or studs should be placed in the mounting pad equally spaced on a bolt circle of 8.00 inches as shown in the above sketch. The bolts should extend at least 2.25 inches above the top of the mounting pad. In pouring the concrete pad, provide a top mounting surface which is flat and level.

The Impact Tester must be truly vertical to assure accurate and reproducible test results. Check installation carefully with a level or plumb bob and shim and grout as required to achieve a true vertical position of the unit.

Although the scale is adjusted at the zero height prior to delivery, it is advisable to check this setting after installation. This is done by placing any one of the die cup assemblies in the cavity of the die cup support, as shown on Figure 1. With the plummet retained in the release platform, lower the platform gradually until the plummet striker contacts the vented plug of the die cup assembly. The height indicator for this condition should read zero. If an adjustment is necessary, it is accomplished by simply loosening the screws which retain the pointer and sliding the pointer up or down in its slotted holes to the zero setting on the scale.

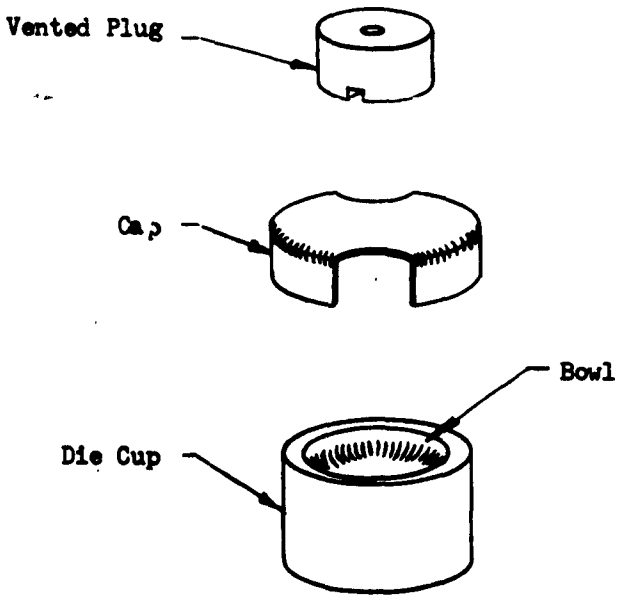
To remove the die cup support for cleaning, raise the one guide rod which is not fastened to the top plate. (Note: Chamfered bore in base plate which distinguishes the loose rod.) Lift the die cup support from the base plate and withdraw same from the tester. Removal and replacement of the plummet assembly to increase or reduce impact energy, as dictated by drop test patterns, is similarly accomplished. Release the plummet from the platform and withdraw same past the clearance provided by the raised guide rod. Lifting of the loose rod can be facilitated by balancing with a counterweight. A tapped hole is provided in the rod for this purpose.

III. OPERATING PROCEDURE

A. General

For general operation of the Impact Tester in an ambient air atmosphere, the procedure is extremely simple and provides a capability of over 40 impact drops per hour. Subsequent sections of this instruction bulletin define the procedures to be employed for impact testing of liquids and/or solids in the presence of cryogenic and other oxidizing media. Also covered are instructions for calibration of the Impact Tester, conversion of drop height to impact energy and recognition of a detonation.

The die cup assembly which holds the sample to be tested consists of three basic parts as follows:



DIE CUP ASSEMBLY
for
Liquid Oxygen Testing

Two different die cup assemblies are supplied with the impact tester. The die cup assembly to be used is dependent upon the material to be tested and/or the surrounding media. Proper selection of the die cup assembly is covered under the pertinent heading in these instructions.

Test specimens are sized to occupy $\frac{1}{2}$ the volume of the die cup bowl. Liquid samples can be carefully measured with a hypodermic syringe; solid samples should be disc-shaped 13/64" in diameter and .010 thick.

With the die cup installed in the recess of the die cup support, place the sample to be tested in the bowl of the die cup, cap the die cup making certain that the die cap is snugly in place, now place the vented plug, slot down, on top of the die cap. Proper center positioning of the vented plug is facilitated by the preformed circular depression in the die cap.

The die cup assemblies (-53) and (-54) are expendable items. Do not re-use any part of the die cup assembly since each piece is distorted during an impact test whether or not a detonation results. Re-used die cup parts will not give reproducible test results.

The impact test is then performed by positioning and locking the plummet release platform with the plummet at the desired height read directly on the scale located to the right of the platform. To release the plummet, depress the control button and the plummet will drop on the die cup assembly driving the vented plug into the test sample thus imparting impact energy to the sample.

At each height a test series of no more than ten trials or drops is performed. If the sample detonates before the tenth drop, no more trials need be made at that height. The threshold valve is defined as the point where no detonation occurs in a test series of ten trials if a single detonation was observed in a test series at one inch higher.

The fastest method of finding the shock sensitivity is to start with the plummet at 36 inches high and conduct a series of drop tests. If a detonation occurs within 10 drops, the plummet is then moved down to $\frac{1}{2}$ the initial height and the test series is performed again. If no detonation occurs, the plummet is moved up to $\frac{1}{2}$ the distance remaining between the two tests. The plummet is always moved in increments of half the remaining distance, until the distance is one inch. The direction of motion is always DOWN if a detonation occurs and UP if no detonation occurs.

For example, a drop height of 36 inches is used and the sample detonates. The second test series is conducted at 18 inches and there is no detonation. Five more consecutive trials reveal no detonation, but on the seventh trial at 18 inches a detonation does occur. The next test series is made at 9 inches and no detonation is obtained. The next test series is conducted at 13 inches where a detonation occurs. The next test series is performed at 11 inches with no detonation. The final test series is made at 12 inches with a detonation. The threshold is then defined at 11 inches.

B. Conversion of Drop Height to Impact Energy

The impact energy imparted to a test sample is dependent upon the weight of the plummet and is proportional to the height from which the plummet is dropped. A curve (Figure 2) is included at the end of these instructions which relates the drop height to the theoretical impact energy in ft/lb per square inch. The energy values are based on a 2000 gram weight for the brass plummet and a 500 gram weight for the aluminum plummet. Manufacturing tolerances permit a weight variation of only ± 0.20 percent for either plummet. The vented plug which is driven into the test sample to impart impact energy is held to a diametral tolerance of ± .001 inch. To calculate the threshold value as impact energy in ft-lbs/sq in for the Reaction Motors impact tester, the following formulae apply:

(1) E (for brass plummet) ≒ 11.55 x drop height (in.)
(2) E (for aluminum plummet) = 2.89 x drop height (in.)

C. Cleaning

Dirt particles, chips or other foreign matter between the bearing surfaces of the die cup support and the base and/or between parts comprising the die cup assembly will absorb energy on impact and adversely affect test reproducibility. These parts, therefore, should be carefully inspected periodically and cleaned with trichlorethylene prior to each series of tests. The plummet striker and the three vertical rods in the vicinity of the impact area should be similarly cleaned. Chips from the detonation should be removed after each test. Your impact tester is cleaned prior to assembly and packaging for delivery. The expendible die cup assemblies should be cleaned before use to assure accurate results. In the interest of accuracy and reproducibility of test results, it is important that critical components of the impact tester be maintained in a hospital clean condition. Die cup assemblies are cleaned in high purity trichlorethylene. The cavity in the die cup and the underside of the cap should be wiped clean with a cotton swab or Q-Tip after removal from the trichlorethylene and just prior to use. Parts may be left to soak in the cleaning fluid until ready for use. However, the parts must be dry. The holes in the vented plugs must be clear. Before test, ultrasonic cleaning can be used, if available. If not, the parts can be gently shaken in the cleaning solution.

D. Impact Testing Liquids

To test a liquid propellant, oil, or other fluids, use a hypodermic syringe to establish volume of test fluid required to fill a clean die cup to brim level. Empty the die cup, clean it thoroughly with trichlorethylene and dry. Place one-half of the above number of drops in the cup, install the die cap over the cup and center the vented plug on top of the cap with the slot down. If the test is to be conducted with room temperature liquids (such as H_2O_2 or acid), the die cup assembly (-53) with the full aluminum cap should be used.

Impact testing in a surrounding of liquid or gaseous oxygen or any cryogenic oxidizer is conducted in the same manner with the following exceptions:

(1) Use the (-54) die cup assembly with the scalloped die cap.

(2) Having placed the die cup assembly with the prepared sample in the die cup support, install the plastic container (-12) on the die cup support as shown on the Impact Tester Assembly (Figure 1).

The plastic container must be used in tests with cryogenic fluids to retain the liquid oxygen around the die cup assembly. After liquid oxygen is poured into the container to a level of approximately ¼ inch above the vented plug, a twenty (20) second cooldown period should be allowed to lapse prior to dropping the plummet. Liquid oxygen must be added to maintain the level. Caution must be taken to use only samples which are more dense than the liquid oxygen so that the sample does not float.

The anvil should not be allowed to get too cold as this will slow the testing. When the temperature of the anvil approaches liquid oxygen temperature, the LOX starts to boil so violently that it is almost impossible to retain enough LOX to properly run the test. When this happens, the anvil should be removed from the machine and allowed to warm up for 20 - 30 minutes at room temperature. (CAUTION: DO NOT heat in furnace as this can crack the anvil). Testing, however, could proceed if a spare anvil were available during the warm up period.

The scalloped die cap is also to be used in tests involved with a gas which must be passed over the sample. A special accessory container will be available shortly for this application.

E. Impact Testing Solids

Solid samples such as plastics, metals, etc., are prepared by filling an estimated one-half volume of the die cup with the solid material. The solid sample should be disc-shaped 13/64" in diameter by .010 thick. To test the sample in liquid oxygen or atmospheres other than ambient air, place the sample in the die cup, use the scalloped aluminum cap and center the vented plug in the cap depression. Follow the normal liquid oxygen test procedure described in Section D. For shock sensitivity testing in an atmosphere of hydrogen peroxide or similar room temperature oxidizer, the sample is placed in the cup and the remainder of the cup is filled with hydrogen peroxide. The aluminum cap without scallops is used.

F. Calibration

The impact tester should be calibrated periodically to ensure consistency in test results. Any impact sensitive material may be used as a calibration sample as long as its properties are consistent from sample to sample. A mixture of Halocarbon 4-11V oil and Union Carbide UCON LB-65 oil is used as a calibration standard. A curve of the impact threshold values of the

oil mixture is included in these instructions (Figure 3). A practical calibration method provides a check on the threshold values in liquid oxygen at three oil volume ratios of 20%, 50%, and 70%. Each sample is measured to about $\frac{1}{2}$ of the volume necessary to fill the die cup and the impact test proceeds as described in Section D for testing in a liquid oxygen atmosphere.

If the impact threshold values for the calibration oil do not agree with the curve values, the Impact Tester should be checked for proper installation or wear, scratches, or particles on any of its parts which may effect the impact results. Furthermore, the mixture should be prepared just prior to use.

The mixture must be stirred frequently to assure homogeniety, as this may affect results. Furthermore, there is some small variation in the shock sensitivity of different batches of fresh calibration fluid.

The accuracy of the machine is $\pm$ 11.55 ft-lb/in^2 or $\pm$ one (1) inch of drop height with the 2 Kg plummet.

As will be seen in Figure 3, the calibration curve is a straight line between the 20% and 70% points. Because of variations in the oil, the 10% and 80% points are sometimes on the line and other times off the line. For this reason, the curve is shown as dotted. The plotted data are shown for the same oil in two different machines. In the 20% to 70% region it was found that to date the oil variations have been within the accuracy of the machine.

IV. RECOGNIZING A DETONATION

Most impact detonations are audibly recognized and are not confused with the normal sound of the plummet striking the die cup assembly. However, muffled detonations are common when the sample is a water base material, such as water-based glycols or alcohol. Testing on a humid day will often cause similar problems when water vapor collects on the sample, in the die cup, or freezes in the liquid oxygen surrounding the sample. If the test operator is in doubt whether the impact caused a detonation, he should inspect the bowl of the die cup for a black residue which is characteristic of a reaction between test sample and oxidizer, or for other evidence of combustion.

APPENDIX IV

RMD IMPACT TESTER EVALUATION AT SwRI

During the Impact Sensitivity Cooperative Test Program No. 2 meeting, held at SwRI in November 1960, the RMD impact tester was brought to the attention of the cooperating laboratories. It was decided by ASD that an evaluation of the tester would be conducted at SwRI with the cooperation of RMD. It was further agreed that SwRI would use the RMD impact tester to obtain data for the Impact Sensitivity Cooperative Test Program No. 3 provided the calibration of the tester, using RMD supplied calibration fluids, proved satisfactory.

An RMD impact tester was loaned to SwRI by RMD. The tester was installed on a 16 × 16 × 24 in. concrete pad in the manner recommended in the operating instructions (see Appendix III) received with the tester.

The test samples used in the calibration tests were mixed from two base fluids, Halocarbon 4-11V and UCON 65-LB, which were supplied by RMD. Three different sample mixtures, 20, 50 and 70 percent by volume of Halocarbon 4-11V, were used. Two series of tests were made on each of the three mixtures of calibration fluids in strict accordance with the procedure outlines in Appendix III. The threshold values obtained in these tests are plotted in Figure 1 in comparison with the accuracy limits specified by RMD. It will be noted that the threshold values of the three mixtures were quite varied and that all of the values obtained by SwRI were outside the specified accuracy limits of the tester.

In the above tests, the vented plug was positioned on the aluminum cap in such a way that the slot of the plug was facing downward and the position of the slot relative to the cap was random, in conformance with Appendix III. However, it was noticed during these tests that the position of the slot on the vented plug relative to the centerline of the cap appeared to have some effect on the threshold values. Tests were then conducted to determine the effect of slot positioning on the threshold values for the different mixtures. Two relative slot positions were used: one parallel to the centerline of the cap and one perpendicular to the centerline of the cap. The respective results obtained by using these test configurations are shown in Figures 2 and 3. These results indicate that the specified limits were not achieved in either case and that lower threshold values were obtained when the slot was positioned parallel to the centerline of the cap in comparison to the case when the slot was positioned perpendicular to the centerline of the cap.

RMD was consulted about the above situation and advised of the finding in regard to the effect of the position of the slot on the threshold value of the sample being tested. A revised test procedure (RMD Specification 7491) was subsequently forwarded by RMD in which the slot on the vented plug was specified to be parallel to the centerline of the cap covering the die cups and an improved vented plug made of a harder material was recommended. Using this revised test procedure and the recommended vented plug, calibration

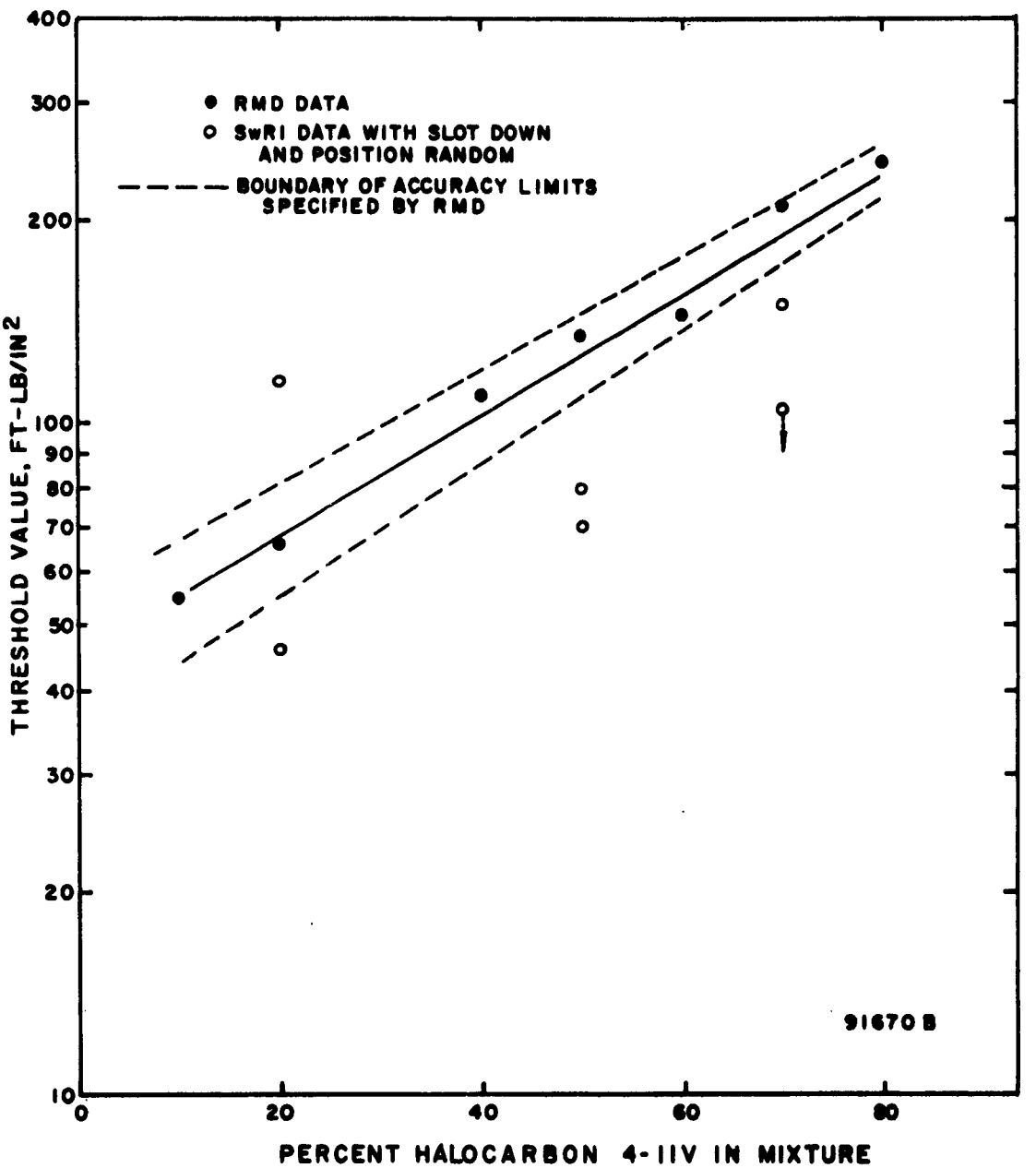


FIGURE 1. COMPARISON OF RMD RESULTS WITH SwRI RESULTS OBTAINED WITH SLOT POSITION RANDOM

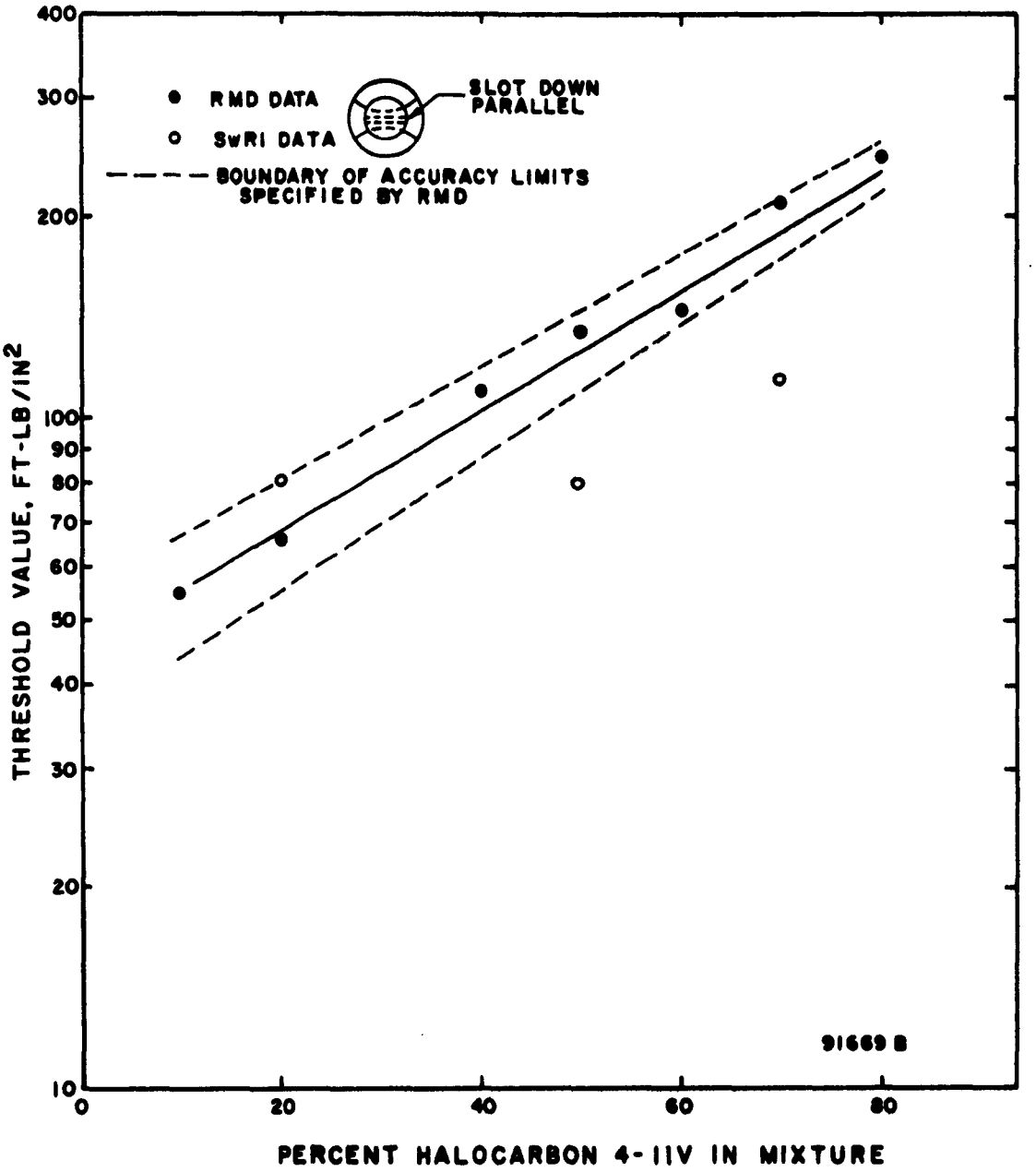


FIGURE 2. COMPARISON OF RMD RESULTS WITH SwRI RESULTS OBTAINED WITH SLOT PARALLEL TO CAP

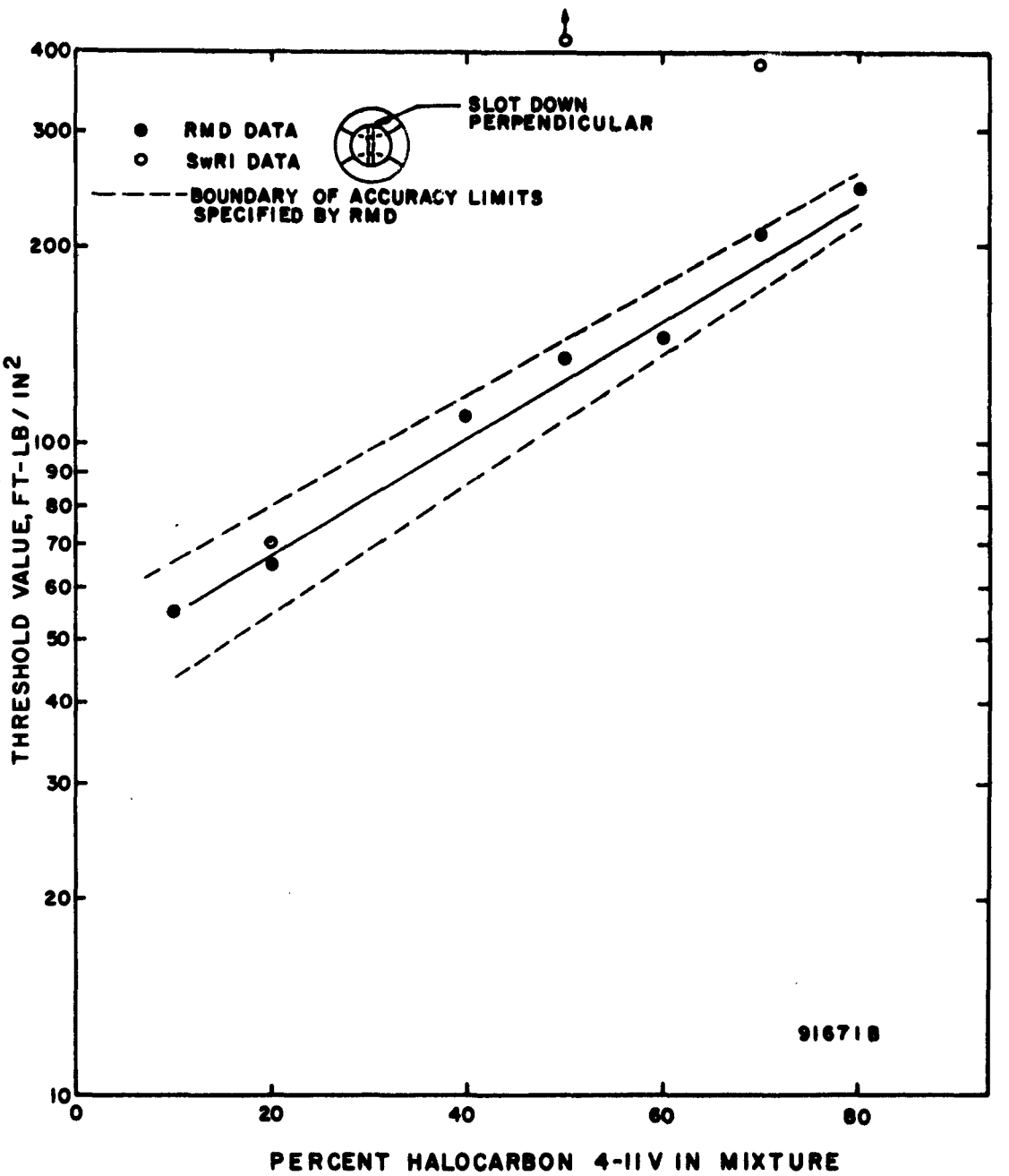


FIGURE 3. COMPARISON OF RMD RESULTS WITH SwRI RESULTS OBTAINED WITH SLOT PERPENDICULAR TO CAP

tests on the RMD tester were resumed at SwRI. Results of these tests are presented in Figure 4. Only one determination on the 20 percent 4-11V was within the limits. Six other determinations (on three different sample mixtures) were located outside the specified limits.

Further consultation with RMD led to the question of interpretation of the criteria of reaction. The section related to the definition of a detonation in Appendix III reads, "Most impact detonations are audibly recognized and are not confused with the normal sound of the plummet striking the die cup assembly. However, muffled detonations are common when the sample is a water base material... If the test operator is in doubt whether the impact caused a detonation, he should inspect the bowl of the die cup for a black residue which is characteristic of a reaction between test sample and oxidizer, or for other evidence of combustion." It was learned from RMD that reactions or detonations were acknowledged only by audible reports in their calibration tests. Flashes or char marks (black residues) by themselves were not considered as evidence of detonations without concurrence of audible reports. The above-quoted definition of a detonation had been interpreted at SwRI prior to this consultation to mean that an audible report and/or a char mark was evidence of a detonation. Since the calibration tests conducted previously were conducted on the basis of this interpretation, eight additional series of tests were made on the three reference fluids in accordance with the clarified interpretation of reaction criteria. The threshold values determined in these eight series of tests are plotted in Figure 5. Again only one determination was within the accuracy limits, while the other seven determinations were outside the limits.

In an effort to find the cause of the deviations in the calibration test results at SwRI, a RMD representative visited SwRI in May 1962 to check the test procedure used and other related test conditions. Several series of calibration tests were conducted at SwRI in the presence of the RMD representative. The cleaning and test procedures were considered satisfactory by the RMD representative. However, two suggestions were made by the RMD representative: (1) It was pointed out that LOX should be added continuously to maintain the liquid level at 1/4 in. above the vented plug during the 20-second cooldown period prior to each test drop. (2) With respect to the definition of a detonation, a reaction should be disregarded if the vented plug does not strike the center of the die cup, even though a positive audible detonation is recognized.

Six series of tests were conducted on samples composed of 50, 70 and 80 percent Halocarbon 4-11V by the RMD representative and the SwRI personnel. The results of these tests are presented in Figure 6. These tests were made at drop heights corresponding to the anticipated threshold values of the respective samples in order to cut down the required test time. It will be noted in Figure 6 that although exact threshold values were not determined in these tests, the potential values were all outside the specified limits of accuracy.

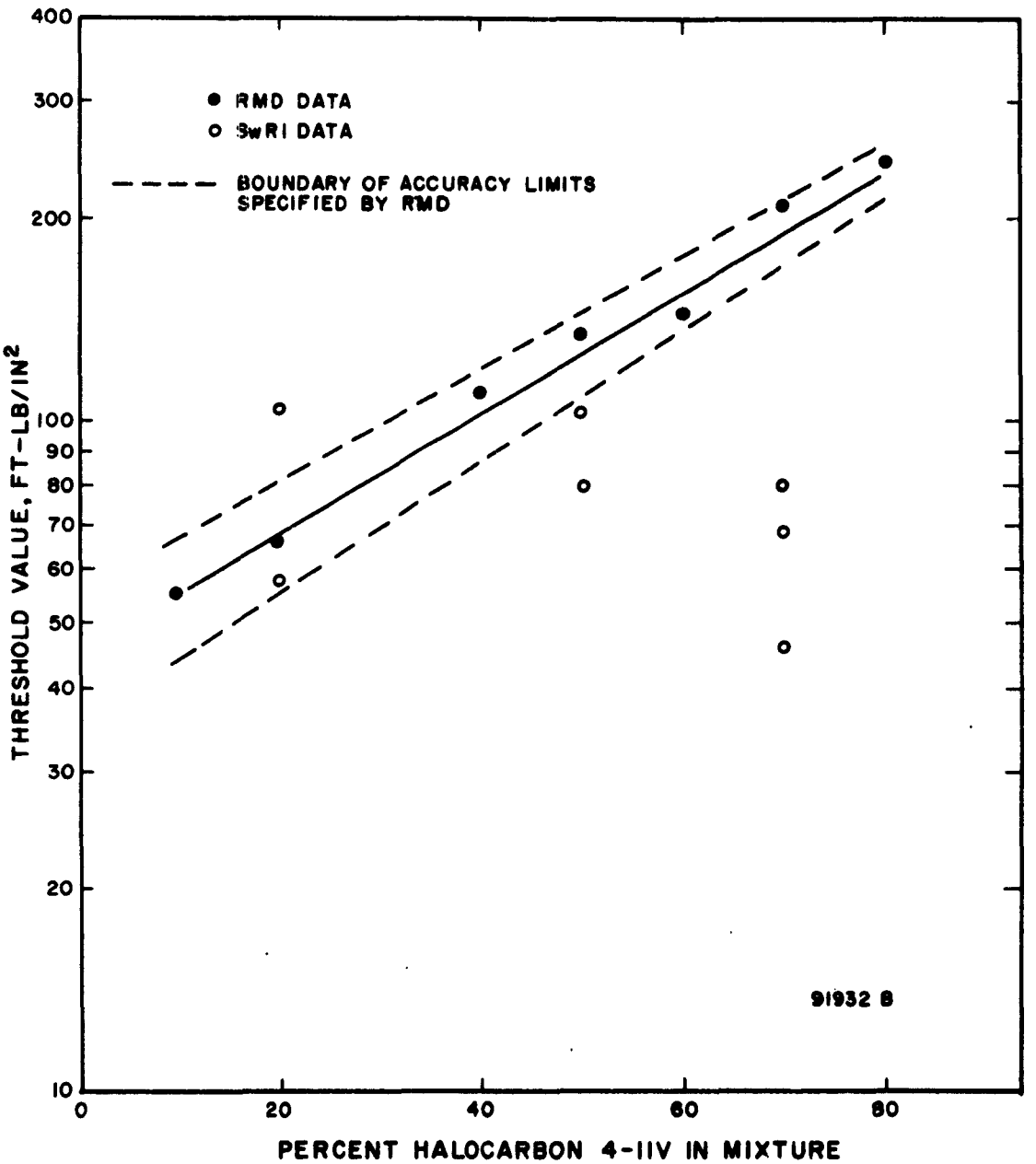


FIGURE 4. CALIBRATION TEST RESULTS ON RMD TESTER USING REVISED TEST PROCEDURE AND IMPROVED PLUG

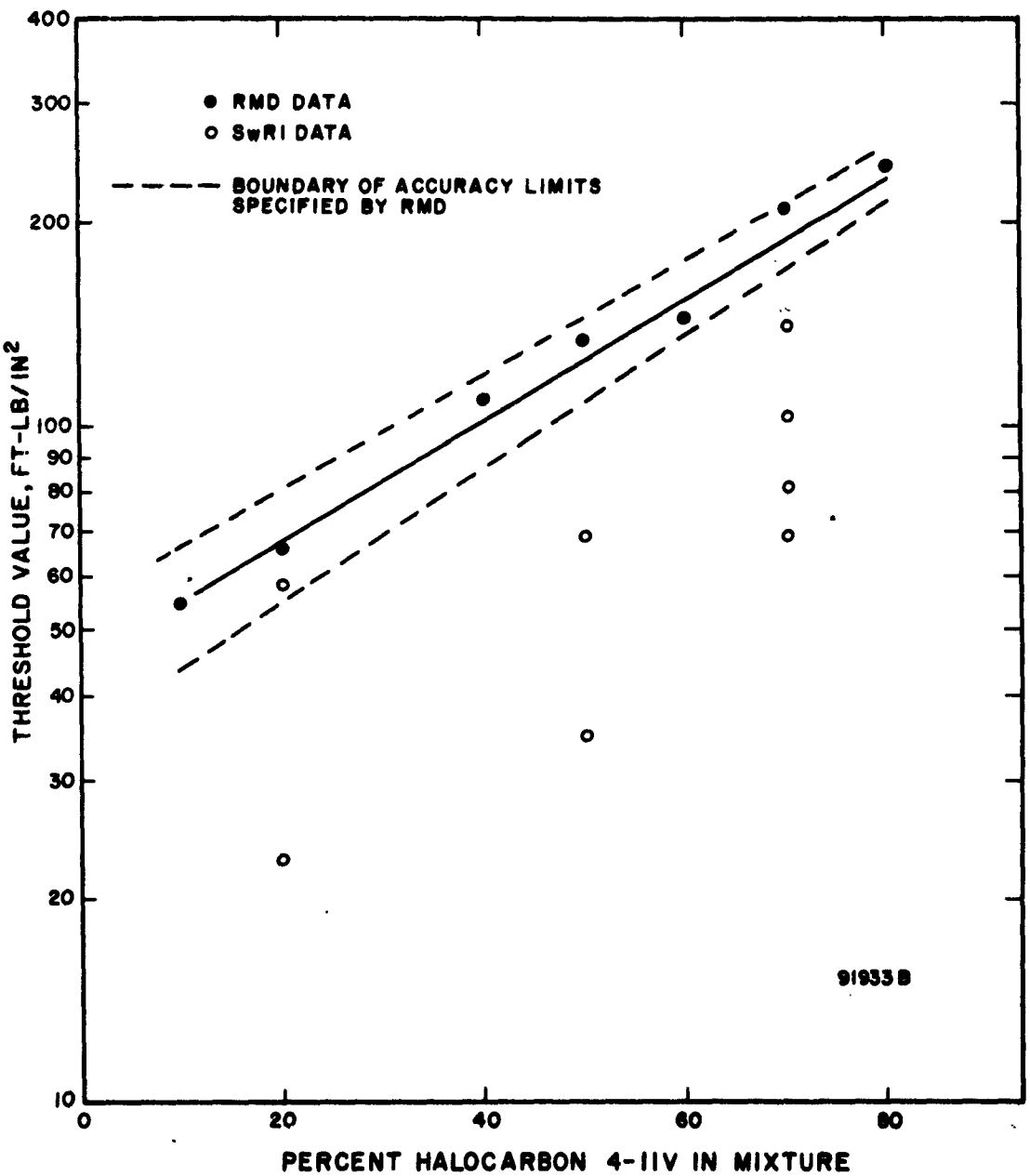


FIGURE 5. CALIBRATION TEST RESULTS WITH ONLY AUDIBLE REPORTS CONSIDERED AS REACTIONS

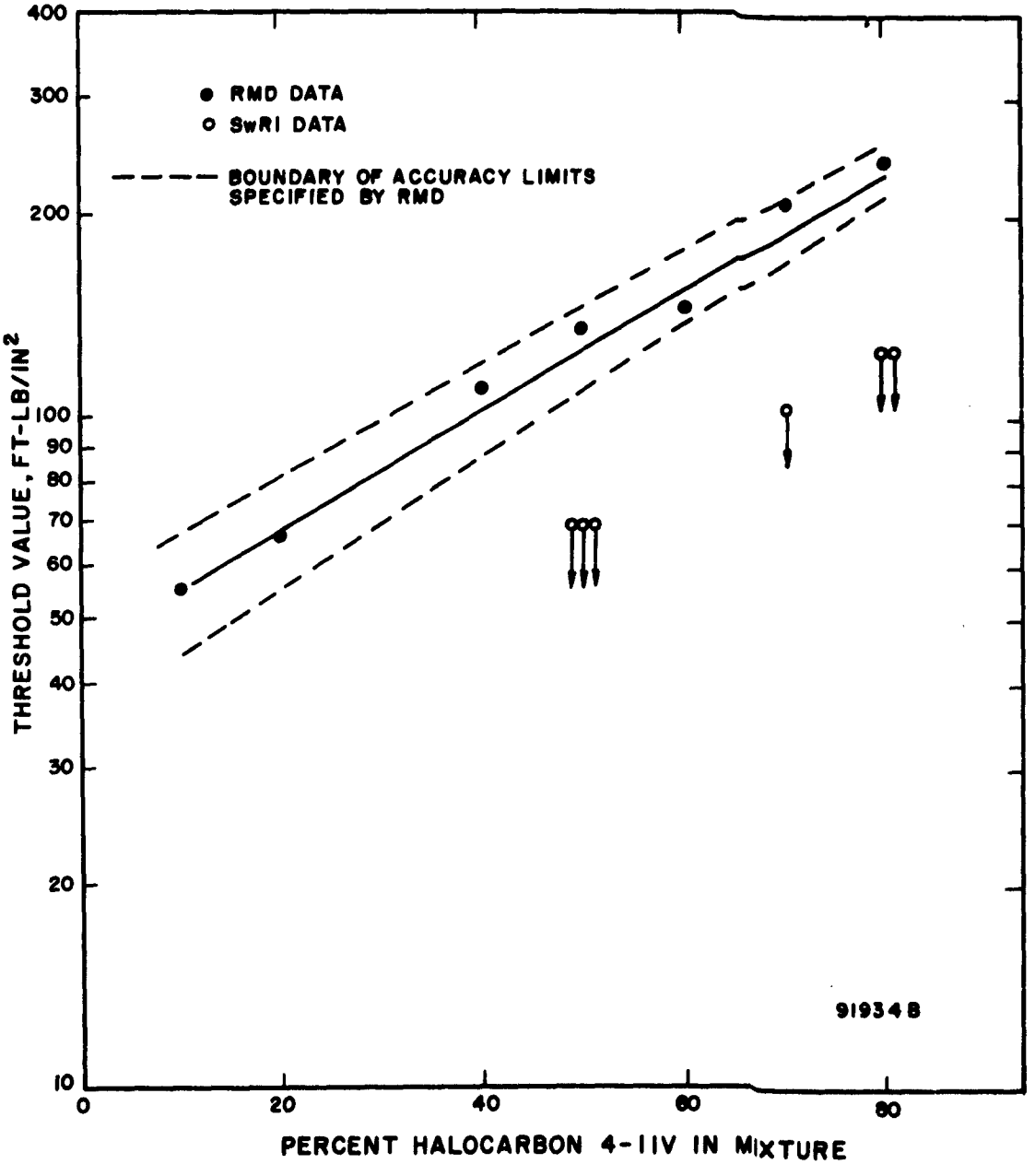


FIGURE 6. CALIBRATION TEST RESULTS IN ACCORDANCE WITH PROCEDURE SUGGESTED BY RMD REPRESENTATIVE

Using 100 percent Halocarbon 4-11V as sample, four drop tests were then made from a drop height of 36 in. and three reactions were observed. Since Halocarbon 4-11V is considered by RMD to be quite insensitive to impact in LOX, this unexpected result suggested the need for blank tests. Sixteen blank tests were subsequently made from a drop height of 36 in. and four flashes were observed, one of which was accompanied by an audible report.

Recent consultations with RMD indicate that a newly designed die cup assembly is currently being tested in an effort to eliminate the problems encountered with the calibration of the tester at SwRI.